

WL-TR-93-3020

ATTACHMENT TECHNIQUES FOR HIGH  
TEMPERATURE STRAIN



STEPHEN P. WNUK, JR.

HITEC PRODUCTS INC.  
100 PARK STREET, UNIT 12  
P.O. BOX 790  
AYER MA 01432

JAN 1993

FINAL REPORT FOR 11/01/90-01/31/93

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION IS UNLIMITED.

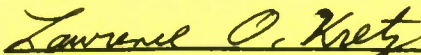
FLIGHT DYNAMICS DIRECTORATE  
WRIGHT LABORATORY  
AIR FORCE MATERIEL COMMAND  
WRIGHT PATTERSON AFB OH 45433-7562

## NOTICE

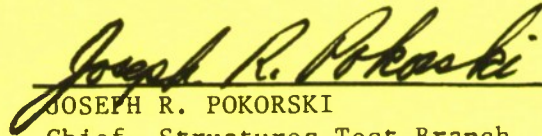
When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely Government-related procurement, the United States Government incurs no responsibility or any obligation whatsoever. The fact that the government may have formulated or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication, or otherwise in any manner construed, as licensing the holder, or any other person or corporation; or as conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

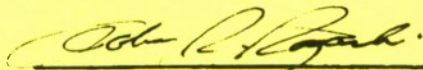
This technical report has been reviewed and is approved for publication.



LAWRENCE O. KRETZ, Project Engineer  
Instrumentation Section  
Structures Test Branch



JOSEPH R. POKORSKI  
Chief, Structures Test Branch



JOHN R. ROGACKI, LT COL, USAF  
Chief, Structures Division  
Flight Dynamics Directorate

If your address has changed, if you wish to be removed from our mailing list, or if the addressee is no longer employed by your organization please notify \_\_\_\_\_, WPAFB, OH 45433-\_\_\_\_\_ to help us maintain a current mailing list.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.



REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE January 1993	3. REPORT TYPE AND DATES COVERED Final Report Nov 90 - Jan 93		
4. TITLE AND SUBTITLE  Attachment Techniques for High Temperature Strain		5. FUNDING NUMBERS C - F33615-90-C-3215 PE - 65502 PR - 3005 TA - 40 WU - 97		
6. AUTHOR(S)  Steve P. Wnuk, Jr.				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) HITEC Products, Inc. 100 Park St, Unit 12 P O Box 790 Ayer MA 01432		8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Flight Dynamics Directorate Wright Laboratory Air Force Materiel Command Wright-Patterson AFB OH 45433-7562		10. SPONSORING / MONITORING AGENCY REPORT NUMBER  WL-TR-93-3020		
11. SUPPLEMENTARY NOTES  This is a Small Business Innovative Research Program, Phase II				
12a. DISTRIBUTION / AVAILABILITY STATEMENT  Approved for public release; distribution is unlimited		12b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) Attachment methods for making resistive strain measurements to 2500°F were studied. A survey of available strain gages and attachment techniques was made, and the results are compiled for metal and carbon composite test materials. A theoretical analysis of strain transfer into a bonded strain gage was made, and the important physical parameters of the strain transfer medium, the ceramic matrix, were identified. A pull tester to measure pull-out tests on commonly used strain gage cements indicated that all cements tested displayed adequate strength for good strain transfer. Rokide flame sprayed coatings produced significantly stronger bonds than ceramic cements. An in-depth study of the flame spray process produced simplified installation procedures which also resulted in greater reliability and durability. Application procedures incorporating improvements made during this program are appended to the report. Strain gages installed on carbon composites, Rene' 41, 316 stainless steel, and TZM using attachment techniques developed during this program were successfully tested to 2500°F. Photographs of installation techniques, test procedures, and graphs of the test data are included in this report.				
14. SUBJECT TERMS  High Temperature Strain Gages, Attachment Techniques, Ceramic Cements, Flame Spray			15. NUMBER OF PAGES 146	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT  Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE  Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT  Unclassified	20. LIMITATION OF ABSTRACT  UL	

## TABLE OF CONTENTS

1.0	<u>SUMMARY</u>	1
2.0	<u>INTRODUCTION</u>	2
2.1	<u>PURPOSE</u>	2
2.2	<u>CURRENT STATE OF THE ART</u>	2
2.2.1	<u>Carbon Composites</u> (Uncoated)	3
2.2.2	<u>Carbon Composites</u> (SiC Coated)	3
2.2.3	<u>Rene' 41</u>	3
2.2.4	<u>TZM</u>	4
2.3	<u>TECHNICAL APPROACH</u>	4
2.3.1	<u>Carbon Composites - Survey</u>	4
2.3.2	<u>Modes of Failure Study - New Gage Design</u>	4
2.3.3	<u>Rene' 41</u>	4
2.3.4	<u>TZM</u>	5
2.4	<u>DESCRIPTION OF PROGRAM EFFORT</u>	5
2.4.1	<u>Carbon Composites</u>	5
2.4.2	<u>Rene' 41</u>	5
2.4.3	<u>TZM</u>	6
3.0	<u>TECHNICAL DISCUSSION</u>	7
3.1	<u>LITERATURE SURVEY COMPILATION</u>	7
3.2	<u>HIGH TEMPERATURE ADHESIVE REQUIREMENTS</u>	7
3.3	<u>COMMERCIAL COATING TEST METHODS</u>	11
3.4	<u>LEAKAGE TO GROUND MEASUREMENTS</u>	11
3.5	<u>THEORETICAL ANALYSIS OF STRESSES WITHIN A HIGH TEMPERATURE STRAIN GAGE INSTALLATION</u>	11
3.6	<u>LEAD WIRE PULL-OUT TEST EQUIPMENT</u>	17
3.6.1	<u>Development of the Micropull Tester</u>	21
3.6.2	<u>Sensing Beam Design</u>	22
3.6.3	<u>Bond Specimen Design</u>	22
	Bonding problems and sources of error	22
3.6.4	<u>Pull Testing Leads Bonded to Glass Specimens</u>	24
3.6.5	<u>Observation of Failure Modes</u>	24
3.6.6	<u>Room Temperature Pull Tests</u>	26



3.6.6.1	<u>Pull Tests on Ceramic Cements and Flame Sprayed Coatings</u>	26
3.6.6.2	<u>Effect of Cement Age on Bond Strength.</u>	29
3.6.6.3	<u>Effect of Cure Temperature on Bond Strength.</u>	29
3.6.6.4	<u>Effect of High Temperature Cure on Cement Strength</u>	29
3.7	<u>THEORY OF ADHESION</u>	33
3.7.1	<u>High Temperature Bonding</u>	33
3.7.2	<u>Ceramic Cements</u>	34
3.7.3.1	<u>Bonding Gages to Glass</u>	34
3.7.3.2	<u>"Lock-up" Cure Temperature of Ceramic Cements</u>	35
3.7.4	<u>Silicone Contamination Studies</u>	37
3.7.4.1	<u>Tape Quality Control</u>	37
3.7.4.2	<u>Cure Temperature vs. Tape Residue</u>	37
3.7.4.3	<u>Silicone Removal</u>	38
3.7.4.4	<u>Preventing Silicone Contamination During Gage Bonding.</u>	39
3.7.4.4.1	<u>General Rules</u>	39
3.7.4.4.2	<u>Ceramic Cement Installation</u>	39
3.7.4.4.3	<u>Flame Spray Installations</u>	40
3.7.5	<u>Wire Temperature During Flame Spraying</u>	43
3.7.5.1	<u>Sample Calculation</u>	45
3.7.5.2	<u>Vortex Cooling</u>	46
3.7.5.3	<u>Temperature Measurement During Spraying</u>	46
3.7.5.4	<u>Coating Thickness</u>	48
3.7.5.5	<u>Uncoated Carbon Composites</u>	48
3.7.5.5.1	<u>Strain Gage Ceramic Cements</u>	48
3.7.5.5.2	<u>Modes of Failure Dictate Gage Design</u>	48
3.7.5.5.3	<u>Convoluteds Leads</u>	50
3.7.5.5.4	<u>Low Expansion Cements</u>	50
3.7.5.6	<u>Flame Sprayed Coatings</u>	50
3.7.5.7	<u>Tests on Carbon Composites</u>	53
3.7.5.8	<u>Silicon Carbide Coated Carbon Composites</u>	55
3.7.6	<u>Rene' 41 Tests</u>	61
3.7.6.1	<u>GPD Low Profile Capacitive Strain Gages</u>	61
3.7.6.2	<u>High Temperature Rene' 41 Tests</u>	64
3.7.6.3	<u>High Temperature Tests on 316 Stainless Steel</u>	70
3.7.6.4	<u>TZM Tests</u>	70
4.0	<u>CONCLUSIONS</u>	80
5.0	<u>LIST OF REFERENCES</u>	81
APPENDIX A		
	Bonding Free Filament Strain Gages with Ceramic Cement	88
APPENDIX B		
	Flame Spray Attachment Procedure	99

## ILLUSTRATIONS

### FIGURES

Figure 1-A	Top View Free Filament Wire Strain Gage . . . . .	10
Figure 1-B	Side View Wire Strain Gage . . . . .	10
Figure 2	Stress Condition for a Wire Bonded to a Strained Surface . . . . .	13
Figure 3A	Tape Carrier Pull Test Specimen Prior to Bonding . . . . .	18
Figure 3B	Pull Test Specimen Ceramic Bonded . . . . .	18
Figure 4	Micropull Tester with Recorder . . . . .	19
Figure 5	Micropull Tester with Capacitive Force Transducer . . . . .	19
Figure 6	Closeup of Micropull Tester . . . . .	20
Figure 7	Closeup of Specimen Clamped to Table and Wire Clamped at Force Transducer . . . . .	20
Figure 8	Closeup of Cam Operated Fine Wire Clamp . . . . .	21
Figure 9	Sketch of Capacitive Sensing Beam Design . . . . .	23
Figure 10	Cross Section of Pull-Test Specimen . . . . .	24
Figure 11	Pull test machine showing microscope setup to view lead wire- ceramic matrix pull-out failures. Lead wires bonded to clear glass slides afford an excellent view of the debonding process . . .	25
Figure 12	Operator observes lead-wire ceramic matrix debonding as pull- out load is applied to the lead. Note the motor drive is not used to apply load while viewing. Instead load is applied by slowly turning a screw-driven slide manually. The X-Y plotter automatically plots applied loads during pullout process . . . . .	25
Figure 13	Closeup of a Lead Wire Bonded to the Test Specimen. The lead wire broke rather than pull out of the cement. The second wire (not shown) pulled out of the cement . . . . .	27
Figure 14	Rokide Bonded Leads, left, 0.005 inch Dia. inch Chromel, Center 0.003 inch Dia. Chromel, Right, 0.003 inch PtNi . . . . .	27
Figure 15	Powder Flame Sprayed FA Powder and 0.002 Dia. Convoluted and Flattened PtNi leads. All lead broke rather than pull out of the coating . . . . .	28
Figure 16	Effect of Cement Age . . . . .	30
Figure 17	Cure Temperature vs. Strength . . . . .	31
Figure 18	Pull Strength of Russian Cement . . . . .	32
Figure 19	Rokide Bonded Strain Gage Attached to Glass Microscope Slide. Viewed from top . . . . .	36
Figure 20	Rokide Bonded Strain Gage Attached to Glass Microscope slide. Viewed from Bottom . . . . .	36
Figure 21	Cross Section of Free Filament Gage Prepared for Flame Spray Installation . . . . .	41
Figure 22	Railroad Tracks Buckled From Heat During Trestle Fire . . . . .	44
Figure 23	Strain Gage Wires Buckle Due to Hot Gasses During Flame Spraying . . . . .	44
Figure 24	Sensor Temperature During Flame Spraying . . . . .	47



Figure 25	A Pt8W strain gage with convoluted leads is positioned on a carbon composite. The composite is coated with 3 to 4 mils of flame sprayed FA powder . . . . .	51
Figure 26	A 5-mil-dia. convoluted type S thermocouple is taped in place next to the strain gage. The gage and thermocouple are boxed in using no. 64 tape to prevent excess over spray on the specimen . . . . .	51
Figure 27	Close-up of Gage and Thermocouple . . . . .	52
Figure 28	Close-up of completed gage and thermocouple installation on carbon composite specimen. Note, extension leads are routed through two hole alumina insulators, which are bonded to the specimen with WC-9 cement . . . . .	52
Figure 29	Not all carbon composites can be coated with ceramics. Powder flame sprayed alumina would not adhere to this specimen . . . . .	54
Figure 30	Powder bonding of a powder flame sprayed alumina coating is shown on this specimen . . . . .	54
Figure 31	Overview of Completed Installation on Carbon Composite Showing Two Hole Alumina Insulators Bonded to the Specimen with WC-9 Cement. Before Test . . . . .	56
Figure 32	Carbon Composite Test Specimen After Plunge Test to 2606°F . . . . .	56
Figure 33	Pt8W Gage and 5-mil convolute Type S Thermocouple Bonded Directly to C/C Using FA Powder Flame Spray. Alumina insulators are bonded with WC-9 cement and Aremco 551A carbon cement . . . . .	57
Figure 34	A 5-mil-diameter convoluted type K thermocouple bonded with WC-9 cement withstands furnace plunge to 2550°F. Note the remains of the thermocouple are a series of small spheres of melted chromel and alumel material . . . . .	57
Figure 35	Silicon carbide coated carbon composite is masked using 3M no. 64 tape. The tape is used to control cement precoat thickness as well as defining the precoat area . . . . .	58
Figure 36	SC cement precoat is applied to silicon carbide coated test specimen . . . . .	58
Figure 37	A spatula is used to uniformly spread the SC cement over the silicon carbide coated surface . . . . .	59
Figure 38	Excess SC cement is scraped off even with the tape surface using a stainless steel spatula. Fast work is required as the cement sets in minutes . . . . .	59
Figure 39	the strain gage and thermocouple are installed using the CR-1000 Mini-Gun and FA alumina flame spray powder. . . . .	60
Figure 40	Completed gage and thermocouple installation on silicon carbide coated C/C. Note the alumina insulators are attached to the specimen using SC cement . . . . .	60
Figure 41	Low Profile GPD Capacitive Strain Gage Spot Welded to Rene' 41 Test Bar . . . . .	62
Figure 42	Capacitive Instrumentation . . . . .	62
Figure 43	Apparent Strain and Total Capacitance vs. Temperature for GPD Capacitive Strain Gage . . . . .	63

Figure 44	Pt8W Gage (left), Pt Resistance Thermometer (center), and Type S Thermocouple (right) Installed With FA Powder Using the CR-1000 Mini-Gun. Initial tack coat is shown . . . . .	65
Figure 45	Close-up of Completed Pt8W Installation on Rene'41 . . . . .	65
Figure 46	Overview of Completed Pt8W Installation on Rene'41 . . . . .	66
Figure 47A	Figure 46 Pt8W Installation After Test. Note specimen melted introducing considerable flow strain into the installation . . . . .	67
Figure 47B	Side View of Melted Specimen . . . . .	67
Figure 47C	Back View of Melted Specimen . . . . .	67
Figure 48	High Temperature Furnace and Controller . . . . .	68
Figure 49	High Temperature Furnace, Controller and Instrumentation . . . . .	68
Figure 50	Resistance vs. Temperature. HDC Dual Element Strain Gage with Pt8W Sensor and ZGS Pt Thermometer . . . . .	69
Figure 51	Chinese 800A Alloy Strain Gage Installed with FA Powder on Rene'41 Bar . . . . .	71
Figure 52	Chinese 800A Alloy Strain Gage After Second Cycle 2500°F . . . . .	71
Figure 53	Resistance vs. Temperature. Chinese 800A Alloy Plunge into 2550° Furnace Specimen Melted on Second Cycle . . . . .	72
Figure 54	FeCrAl Foil Strain Gage Prepared for Installation by Powder Flame Spray. Note the large foil end tabs and lead pads have been cut off and a tape carrier applied. Leads are 5 mil chromel rolled flat in the weld area . . . . .	73
Figure 55	Resistance vs. Temperature. Plunge into 2550°F Furnace Specimen melted on first cycle . . . . .	74
Figure 56	Pt8W Gage on 316 S.S. After Test to 2550°F. Note Beginning of melting under alumina insulators near right end . . . . .	75
Figure 57	Resistance vs. Temperature. Pt8W Alloy Strain Gage . . . . .	76
Figure 58	Resistance vs. Temperature. Pt8W Strain Gage on C/C . . . . .	77
Figure 59	(Top Bar) Ceramic Cement Precoat on TZM after exposure to 1000° for 3 Days . . . . .	81
Figure 60	(Bottom Bar) Weldable Strain Gage Installation on TZM by Spot Welding and Exposed to 1000°F for 3 Days . . . . .	81
Figure 61	(Top Bar) An uncoated TZM specimen begins to "smoke" at 1500°F and grow large crystals on the surface . . . . .	81
Figure 62	(Bottom Bar) TZM Specimen with Precoat Patches of Ceramic Cement Counter Clockwise from Top Left: 1) Yellow Cerro, 2) Yellow Cerro, 3) WC-9, and 4) Red Cerro . . . . .	81

## **TABLES**

Table 1	High Temperature Strain Gages for Uncoated Carbon Composites . . . . .	8
Table 2	High Temperature Strain Gages for Rene'41 . . . . .	9
Table 3	Physical Properties of Common Lead Wires . . . . .	16
Table 4	Maximum Allowance Strain, $\epsilon$ . . . . .	16



## 1.0 SUMMARY

The primary objective of this project was to investigate attachment methods for high temperature strain gages and to develop attachment techniques usable to 2500°F. A secondary purpose was to develop methods for qualifying candidate attachment systems and to produce a reference manual on successful techniques.

High temperature adhesive requirements were examined. A theoretical investigation of strain transfer into the strain gage was made. Since no correlation between properties of cements and their performance in high temperature strain gages has ever been made, a pull tester was designed to simulate strain transfer conditions within a strain gage and to make measurements to predict performance of the bonding medium.

The theory of adhesives, and bonding problems associated with the gage tape carrier were also studied. The pull tester was used to evaluate bond strength of numerous high temperature adhesives. Pull tests were performed on leads bonded to glass microscope slides, and the pull-out process was observed under a stereo microscope.

Silicone tape adhesive contamination was studied in detail and ways to prevent it are documented. Wire temperatures during flame spraying were examined, and a method to keep gage wire cool during flame spraying was developed. Improvements in attachment methods which increase reliability have been made for both ceramic cement and flame spray bonding. Detailed written procedures are appended to this report. Flame spray attachment procedures developed during this program were used to attach various strain gages to:

- A. Rene' 41
- B. Carbon Composite (uncoated)
- C. Carbon Composite SiC Coated
- D. 316 Stainless Steel
- E. TZM

The gage installations were successfully tested for apparent strain to 2500°F.

## 2.0 INTRODUCTION

### 2.1 PURPOSE

The objectives of this program were (1) to survey attachment techniques for high temperature measurements as they apply to specific materials of interest to the Air Force, (2) to develop test methods for qualifying candidate attachment systems, (3) to evaluate selected attachment systems to 2500°F and (4) to produce a reference manual on attachment methods.

### 2.2 CURRENT STATE OF THE ART

Existing resistive strain gages are usable to 1000°F short term static, 2400°F dynamic measurements. Current developments<sup>1</sup> may increase static measurements to 1400°F with future potential in the 1800° to 2000°F range, and dynamic potential to 3000°F.

This project concentrated primarily on resistive strain gages. Although other types are available, resistive strain gages are attractive because of:

- A. small sensor size,
- B. direct attachment,
- C. electrical readout (Gage may be located at inaccessible location - as in a jet engine.),
- D. sensors are durable in hostile environments - often will stay on until part fails, and
- E. low cost.

The resistive gage types commonly employed at high temperature are:

- A. free filament wire
- B. flattened wire
- C. foil and
- D. bonded weldable.

The most common attachment techniques for resistive strain gages are:

- A. ceramic cement bonding
- B. flame spray bonding, and
- C. weldable strain gages attached by spot welding.

The strain gage selection criteria and the method of attachment is governed by test parameters specific to the program and to a large extent on the specimen material. The three specimen materials of interest to the Air Force on this program are



carbon composites, Rene' 41, and TZM. Specimens of each material were supplied by the Air Force for testing in the program. Bonding to each material requires a somewhat different approach.

#### 2.2.1 Carbon Composites (Uncoated)

Several years of development effort directed primarily to strain measurements on carbon composites used in rocket nozzles are reported in Reference 2. Ceramic cements were successful for short term tests up to 1400°F. Rokide flame spray would not stick to carbon composites, but flame sprayed alumina powder adhered well in tests to 2000°F<sup>3</sup>. WC-9, a high alumina ceramic cement developed in the above program<sup>3</sup>, also adhered well and withstood repeated cycles to 2000°F. Boeing has reported<sup>4</sup> success in attaching capacitive gages to carbon composites for testing in a nitrogen atmosphere.

#### 2.2.2 Carbon Composites (SiC Coated)

The procedures for coated carbon composites are complicated by:

- A) the difficulty bonding to silicon carbide, and
- B) the problems associated with the numerous cracks in the coating severely limits the life of attached instrumentation.

Certain conductive materials were found which would bond to silicon carbide. A procedure was adopted to apply a 4-6 mil precoat of conductive cement, such as LEX-10<sup>5</sup> which bonds well to the silicon carbide. Strain gages are then bonded to the LEX-10 precoat using conventional ceramic cements or flame spray methods. The LEX-10 was limited to about 1500°F. Later, SC cement<sup>6</sup> was found useful as a precoat to 2550°F.

#### 2.2.3 Rene' 41

Bonding to Rene' 41 is straightforward. The majority of high temperature strain measurements in the past have been on super alloys, and attachment techniques are well developed. Ceramic cements, Rokide and powder flame spray, and spot welding are among the common attachment methods used in this program.

#### 2.2.4 TZM

TZM is a high strength molybdenum alloy consisting of 99.4% molybdenum, 0.5% titanium, and 0.08% zirconium. Little is written about attaching strain gages to this material. TZM was found to oxidize severely in air at moderate temperatures, and a protective coating had to be applied. A coating consisting of sodium silicate impregnated Rokide allowed testing to 2600° F for short term.

### 2.3 TECHNICAL APPROACH

#### 2.3.1 Carbon Composites - Survey

Ceramic cements are limited to about 1700°F, Rokide is excellent electrically, but will not stick to carbon composites, powder flame sprayed alumina is even better electrically and adheres well to carbon composites but is difficult to use, and has had very limited usage to date. WC-9, a high alumina experimental cement formulated to work on carbon composites, is useful for bonding terminals and thermocouples and is useful as a structural adhesive.

#### 2.3.2 Modes of Failure Study - New Gage Design

Detailed study of modes of failure on the Thiokol program<sup>2</sup> revealed failures are caused by shear stresses between the gage and cement and the cement and specimen. A mismatch in thermal coefficients of expansion of the various components contributes greatly to these internal stresses. A new gage design and lead system was initiated in this program which would minimize these problems. Data from the pull tester also contributed to the design. A thorough study and test program to optimize flame spray parameters was needed to produce consistent, strong bonds to carbon. Gage temperature during spraying was measured, the use of various cooling schemes were studied as well as the usual controls of gas pressures, flow and spray distance.

The results of the gage design, pull test data, and refinements in flame spraying procedures were tested on carbon composite specimens. WC-9 was tried as a thermocouple bonding medium as well as a structural adhesive to attach ceramic insulators to the carbon specimen.

#### 2.3.3 Rene' 41

The results from the carbon composite work was also applied to the Rene' 41 tests. A variety of state-of-the-art gage types were tested. Two low profile GPD type capacitive strain gages were built and tested to 2000°F on Rene' 41. Chinese 800 alloy, some FeCrAl foil gages (ME-2 type 2104), platinum compensated platinum tungsten dual element gages and an improved platinum



tungsten gage design were to be tested to the melting point of the Rene' 41. A platinum tungsten gage was also tested on 316 stainless steel which melts at 2550°F somewhat higher than the Rene' 41.

#### 2.3.4 TZM

Because of oxidation problems, our choice with TZM was to either test in vacuum, inert gas, or to apply a protective coating for testing in air. Testing in air is more practical in our lab. Bonded patches of ceramic cement were used to determine the temperature limit for uncoated TZM. For tests above 1000°F, it was necessary to develop a protective coating. Once a coating was found, attachment of instrumentation to the coating would be developed.

### 2.4 DESCRIPTION OF PROGRAM EFFORT

Compilation of existing information on strain gages, cements, lead wires, and insulation was tabulated for each material of interest. Candidate adhesives were then further screened on the pull tester. The tester was designed and built to provide quantitative data on the bond strength of adhesives to strain gage lead wires and to the substrate.

#### 2.4.1 Carbon Composites

From the failure mode studies of numerous prior tests on carbon composites, a design philosophy evolved and, combined with pull test data, was applied to the design of the gage and attachment system. A great deal of development effort was expended developing flame spraying procedures with the CR-1000 Mini-Gun<sup>7</sup> and high purity FA alumina<sup>7</sup> powder. Improvements in the gun combustion hardware and in the development of a mini-hopper in the past few years had greatly improved the sprayability of ceramics. On this program, further refinements in the application technique were evaluated: (1) the measurement of sensor temperature during spraying, (2) the use of air cooling, and (3) the use of vortex air coolers were studied to further improve gage installations.

A gage attachment system was developed which increased the upper temperature limit for tests on carbon composites from 1700°F to 2550°F. The strain gage employed a 0.00055 inch diameter Pt-W sensor with 0.002 inch machine convoluted and flattened platinum nickel leads. The type S thermocouple was also machine convoluted.

#### 2.4.2 Rene' 41

Most of the design and development work applied to carbon composites were also tested on Rene' 41. A variety of gage types currently in use or under development were tested on Rene' 41. This included the low GPD profile capacitive gage,

Chinese and domestic FeCrAl alloy gages and the new Pt W gage design used on carbon composites above. The gages were operated within their design range except for the FeCrAl and Pt W gages which were evaluated up to the melt point of the Rene' 41. A Pt W gage, Rokide installed, was also tested up to the melt point of 316 stainless steel, a high expansion material.

#### 2.4.3 TZM

The initial program effort was to develop a coating to protect the TZM from oxidation, and to which strain gages could be bonded. Dip coatings were tried first, followed by sodium silicate cements, ceramic cements, Rokide, and Rokide impregnated with silicates and phosphates. Once a suitable coating was found (a Rokide coating impregnated with sodium silicate), strain gages and thermocouples were applied directly to the coating using the CR-1000 Mini-Gun and FA alumina powder. Because the TZM specimens supplied by the Air Force were large, some 20 mil thick sheets of molybdenum 99% were acquired and used for these tests. Apparent strain tests were run to 2550°F for numerous cycles without failure, and recorded on an X-Y plotter.

### 3.0 TECHNICAL DISCUSSION

#### 3.1 LITERATURE SURVEY COMPILATION

The results of a survey of current gage types and application techniques, useful temperature range, leads and lead insulation is tabulated in tables 1 and 2. Table 1 is prepared for carbon composites, and table 2 for Rene' 41. No prior work could be found for TZM. A reference is given for each line item in the table and should be consulted for further details. Some of the strain measuring systems in the tables were selected for further testing in this program.

#### 3.2 HIGH TEMPERATURE ADHESIVE REQUIREMENTS

Common strain gages involve three basic components, the sensing grid, gage backing and bonding adhesive. High temperature strain gages employ only two components, the sensing grid and the adhesive, which also serves as the gage backing. See Figure 1. Therefore the adhesive becomes the all important component which must bond well to the structure, bond to the strain sensing grid, transfer strain from the structure the sensing grid, and at the same time maintain a high level of electrical isolation between the strain gage and ground over a wide temperature range.

A good high temperature strain gage adhesive should possess the following characteristics:

- A) Bond sufficiently well to materials of interest to faithfully transfer strain from the structure into the adhesive matrix.
- B) Be easy to use
- C) Be capable of 5000 $\mu$  inch total strain
- D) Withstand thermal shock
- E) Bond well to strain sensor wire, and faithfully transfer strain from the matrix into the sensor without failure within the desired strain range
- F) Cure at a relatively low temperature (not to exceed 600°F)
- G) Possess high electrical insulation values over a wide temperature range
- H) The thermal expansion of the adhesive should be compatible with the structural material of interest
- I) The adhesive should not lose strength or creep within the strain and temperature range
- J) The adhesive should be chemically compatible with structural materials and strain sensing grid materials.



Table I High Temp Strain Gages For Uncoated Carbon Composites

TEMPERATURE NDRMAL OPERATING	°F MAXIMUM	SENSDR DESCRIPTION	SENSDR TYPE	ATTACHMENT PROCESS	CURE TIME/TEMP	TYPE OF MEASUREMENT	TERMINALS	TERMINAL ATTACHMENT	CONNECTION MEDIUM	CONDUCTOR	LEAD CABLES INSULATION	LEAD ATTACHMENT	PROTECTION/ SHIELDING	NORMAL	LIFE MAX TEMP	REFERENCE	COMMENTS
550	660 STATIC	HFK-12-260-LCW-0	Resistive K Alloy	Ceramic Cement Only	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600°F	Static/ Dynamic	Alumina Feed-Thru's	Bonded-Carbon Cement R.T. Cure	Brze	N.C.C.	Fiberglass	Bonded Straps Carbon Cement or WC-8 Cement 600°F Cure	None	Month	Day-Static Hrs-Dynamic	69, 78	General purpose. Self compensated strain gage.
	1600 Dynamic		Free Filament														
560	660	HDT-12-260-LCW-0	Resistive K Alloy	Ceramic Cement Only	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600°F	-	-	-	-	-	-	-	-	-	-	69, 78	Strain-temperature sensor. Thermal stress measurement during very high heat rates. Special circuit required
			Dual Element Free Filament														
800	1100	HDA-12-260-SPW	Resistive Dual Element	Ceramic Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 800°F	-	-	-	-	N.C.C.	-	-	-	1 Day	1 Hour	69, 74, 76, 89, 80	For short term testing. Moderate heat rates. Special circuit required.
			Free Filament	Power Flame Spray	None	-	-	-	-	-	-	-	-	-	-		
860	1200	HDB-12-260-SPW	Resistive Dual Element	Ceramic Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600°F	-	-	-	Brze	N.C.C.	-	-	-	1 Day	1 Hour	69, 74, 75, 89, 90	For short term testing. Moderate heat rates. Special circuit required.
			Free Filament	Power Flame Spray	None	-	-	-	Spot Weld	Chromel	Nextel	-	-	1 Day	1 Hour		
900	1300 Est	PdCr/Pt	Resistive Dual Element	FA Powder =4% ZrO <sub>2</sub>	None	-	-	-	Brze	N.C.C.	Fiberglass	-	-	Unknown	Unknown	74, 78, 79, 83, 80	NASA-Lewis/UTRC Experimental
			Free Filament	Power Flame Spray													
1295	1295	HFC-7-12-260-SCW	Resistive Chinese 700° Alloy	Ceramic Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 800°F	Static	-	-	Spot Weld	Chromel	Nextel	-	-	1/2 Day	1/2 Day	74, 77, 81	Chinese 700°C alloy strain gage. Compensated for steel.
			Free Filament	Power Flame Spray	None	-											
1476	1476	HFC8-12-260-SCW	Resistive Chinese 800° Alloy	Ceramic Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600°F	-	-	-	-	-	-	-	-	-	-	74, 77	Chinese 800°C alloy strain gage. Compensated for steel.
			Free Filament	Power Flame Spray		-											
1200	1500	HTC-DC-100-6	Capacitive Half Bridge	Ceramic Pins	R.T.C.	-	None	None	Spot Weld	Chromel Special	Nextel Shielded Coax	-	Inconel Box Radiation	Years	Months	71, 82	Capacitive strain gage.
1600	2000	HTC-DC-100-8PT	Capacitive Half Bridge	Ceramic Pins	R.T.C.	-	-	-	-	-	-	-	-	Years	Hours	70, 71, 82	Experimental capacitive strain gage.
-	2000	2104	Resistive Foil	Powder Flame Spray	None	-	Alumina Feed-Thru's	Alumina Feed-Thru's	-	Chromel	Nextel	-	None	Unknown	Hours	70, 74	Foil strain gage Fe/Cr/Al
-	2000	BCL-3	Resistive Free Filament	Powder Flame Spray	None	-	-	-	-	-	-	Bonded Wc-9 Cement. 600°F Cure	-	-	Unknown	83	Fe/Cr/Al wire gage
-	2660	HFP-12-260-SPW	Resistive Free Filament	Powder Flame Spray	None	Dynamic Only	-	Bonded WC-9 Cement 600°F Cure	-	Pt Rho.	-	-	-	-	1/2 Hour	74, 76, 81	Dynamic measurements above 1800°F.

Table 2 High Temperature Strain Gages For Rene' 41

TEMPERATURE NDRMAL OPERATING	°F MAXIMUM	SENSDR DESCRIPTION	Sensor Type	Attachment Process	Cure Time/Temp	Type of Measurement	Terminals	TERMINAL ATTACHMENT	CONNECTION MEDIUM	LEAD CONDUCTDR	CABLES INSULATION	LEAD ATTACHMENT	PROTECTION/ SHIELDING	NDRMAL	LIFE MAX TEMP	REFERENCE	CDMMNTS
550	550 Static 1500 Dynamic	HFK-12-250-LCW-6	Resistive K Alloy Free Filament	Ceramic Cement Only	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 800° F	Static/ Dynamic	Alumina Feed-Thru's	Spot Weld Straps	Brze or Spot Weld	N.C.C.	Fiberglass	Spot Weld Straps	None	Month	Day-Static Hre-Dynamic	81, 52, 84	General Purpose, Self compensated strain gage.
550	550 Static	3401	Resistive Foil	Ceramic Cement Only	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 800° F	* / *	**	-	**	*	*	**	*	*	*	52, 84, 85	General Purpose, Self compensated strain gage.
800	1100	HDA-12-250-SPW	Resistive  Dual Element	Ceramic  Rokide	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600° F None	Static	**	Spot Weld Straps .	**	*	*	**	*	1 Day	1 Hour	74, 75, 88, 80	For short term temp. Moderate heat rate. Special circuit rate.
850	1200	HD8-12-250-SPW	Free Filament  Resistive  Dual Element	Powder F.S.  Ceramic  Rokide	None  *  *	Static	**	*	** **	*	High Temp Glass or Nextel	**	*	1 Day	1 Hour	74, 75, 88, 90	-
800	1100	PdC/Pt	Free Filament  Resistive  Dual Element  Free Filament	Powder F.S.  Only	None	Static	**	*	**		*	**	*	1 Day Est.	1 Hour Est.	74, 77, 81	NASA-Lewis/UTRC Experimental.
1475	1475	HFC8-12-250-SCW	Resistive  Chressee 800°C Alloy	Ceramic Cement Rokide	*	Static	**	*	**	Chromel	Nextel	**	*	Hours	Hours	74, 77	Short term static tests at constant temperature above 1100°F.
1100	2000	Clip Gage	Full or 1/2 Bridge  Resistive	Spot Weld  Metal Flame Spray	None  None	Static	Alumina Feed-Thru's	Spot Weld	Brze  Spot Weld	N.C.C.  Chromel	Fiberglass  Nextel	Spot Weld Straps  Spot Weld Straps	Inconel Box  .	Days	Hour	87	For high elongating measurements using resistive sensor.
850	1200	SG425	Resistive Weldable	Spot Weld	None	Static	None	None	Integral	Sheathed Copper	MgD	Spot Weld Straps	None	Days	Hours	88	Hermetic gage. Useful in steam on corrosive environments.
1200	1600	HTC-DC-100-6-TC	Capacitive Half Bridge	Spot Weld	*	Static	Integral	Spot Weld	Spot Weld	Inconel 800 Special Coax	Nextel Special	**	Inconel Box	Months Years	1 Month	82, 87	Most stable. Long static measurements. creep measurements.
1500	2000	HTC-DC-100-8-PT	*	Spot Weld	*	Static	**	*	**	*	*	**	*	Month	Hours	70	Experimental
-	2000	2014	Resistive	Ceramic				Spot Weld Straps	**	Chromel	Nextel	**	None	Unknown	Unknown	74, 85	Short term static
			Foil	Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600° F	Static/ Dynamic	Alumina Feed-Thru's										
			Rokide	None													
			Powder F.S.	None													
2000		8CL-3	Resistive	Ceramic Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 800° F			*	**	*	*	**		Unknown	Unknown	74, 83	Short term static
			Free Filament	Rokide	None	-	**						None				
-	1800	H8WAPT	Weldable Resistive	Powder F.S.  Spot Weld	None  None	Dynamic Only	None	Integral	Integral	Inconel C/A	Sheathed MgD	Spot Weld Straps	None	Hours	Hour	85	Weldable. Dynamic only.
-	2475	HFP-12-250-SPW	Resistive  Free Filament	Ceramic Cement	1/2 hr @ 200°F, 1/2 hr @ 400°F, 1 hr @ 600° F		Alumina Feed-Thru's	Spot Weld	Spot Weld	Pt Rho	Nextel	Spot Weld	None	Hours	Hour	52	Dynamic only.
				Rokide	None												
				Powder F.S.	None	-											

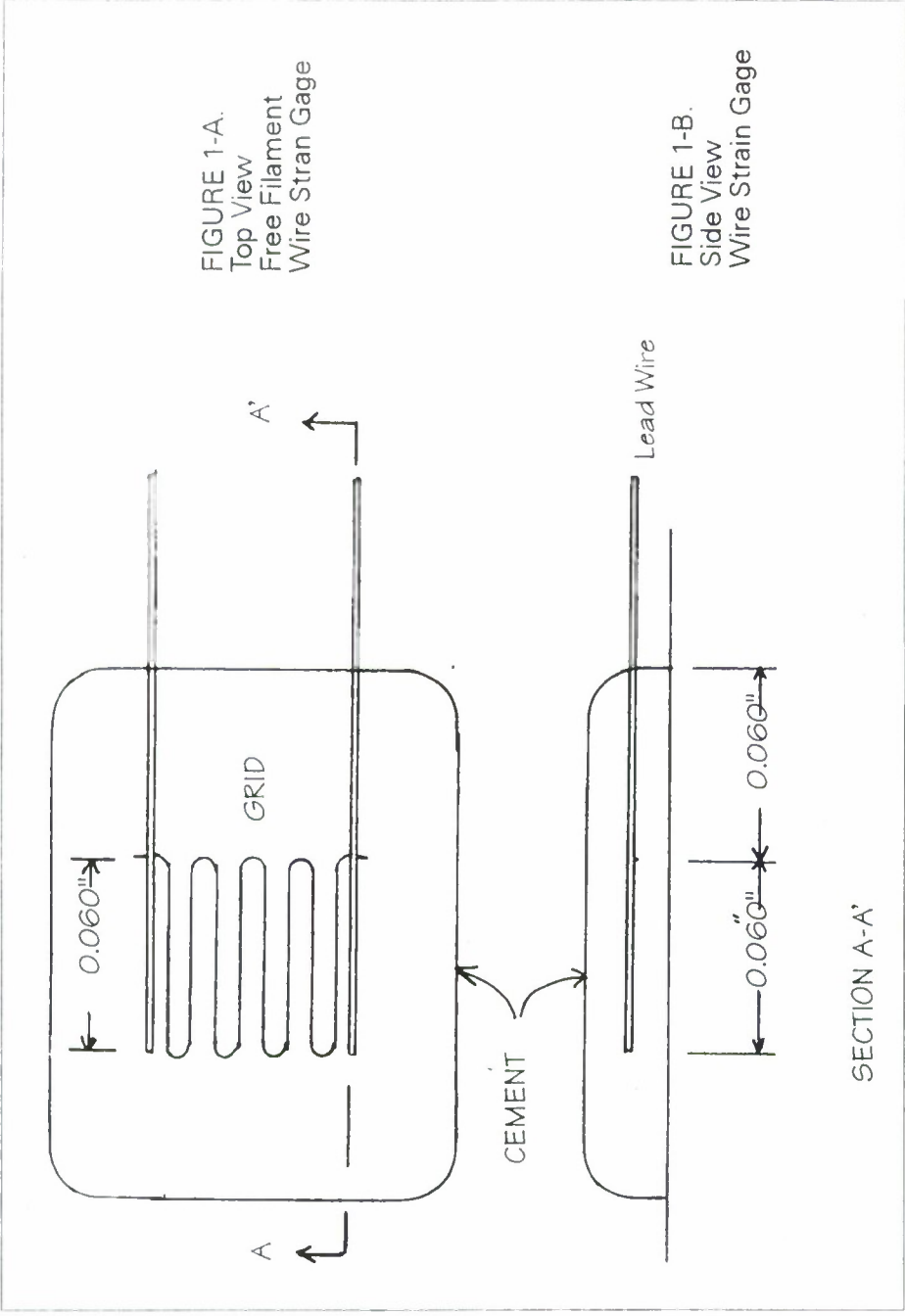


FIGURE 1-A and 1-B. Free Filament Wire Strain Gage, Top and Side Views.



### 3.3 COMMERCIAL COATING TEST METHODS

A search of test methods for testing coatings was made. ASTM C633-79 (Reference 15) for testing flame sprayed coatings seems to be the primary test method in use today. Verbal inquiries posed to various flame spray operators in the field confirmed this. The bending test method used by NBS and reported in ASTM STP-230<sup>8,9</sup> is relatively easy to use. A modified version of this procedure was tried in the lab with poor results. All ceramic cements tried had very low bond strength using this test method, even though the cements were excellent for bonding strain gages.

The important structural properties of a ceramic cement are (a) shear strength in bonds to metals, (b) modulus of elasticity and (c) tensile strength. None of these properties are usually reported. Sermatech<sup>10</sup> does report shear stress values for P1 and PBX strain gage cements. The highest values reported, however, are nearly an order of magnitude lower than measured values from pull tests on strain gage lead wires. Therefore, a more suitable test method for evaluating cements and coatings was needed for evaluating candidate strain gage adhesives.

### 3.4 LEAKAGE TO GROUND MEASUREMENTS

Leakage measurements are an integral part of the information required by the strain gage user. A survey of prior practice was conducted (Reference 8, 9, 10, 11, 12, 13, 14, 15, 16). The ac, dc constant voltage, and d.c. constant current systems were looked at as well as the errors caused by leakage to ground on various strain measurement systems. Results of this study indicate the user should use a measurement system similar to the strain measurement system to be used on the test. From this standpoint errors caused by leakage will be no greater than those indicated by the leakage measurement.

Manufacturers of cements should use an ac measurement system which represents the worst case possible. See Reference 17. A full and complete treatment of this subject is covered in Reference 15.

### 3.5 THEORETICAL ANALYSIS OF STRESSES WITHIN A HIGH TEMPERATURE STRAIN GAGE INSTALLATION

It is necessary to understand the fundamental physical laws governing strain transfer from a structure into the grid of a strain gage. While the concept of a strain gage is relatively simple and well understood, the state of stress within the sensor is very complex. The analysis of stresses and strains within the bonding matrix and the transfer of stresses into the sensing wire or foil have been analyzed by numerous investigators (References 18, 19, 20, 21). The AGARD report by Kottkamp et al<sup>18</sup> also cites extensive theoretical work by Rohrbach and Czaika.

Some of the work reported in Peter Stein's Strain Gage Readings <sup>20</sup> refers to work by Fourethier reported in Analyse Des Constraints, Vol. II. The author has borrowed from extensive work done by researchers, himself included, in the BLH strain gage development lab dating back to the 1960's where a great deal of theoretical, and empirical strain gage design work laid the foundation for today's strain gages.

With all of the theoretical analysis which has been made to date, no ready method has been available which would predict the behavior of a given strain gage adhesive based on the properties of the adhesive. The theoretical analysis, however, contributes to a better understanding of the interaction of the various physical properties in strain gage operation.

To date, there is no correlation between measured bulk properties of adhesives and their performance in high temperature strain gages. One purpose of this program was to devise a test apparatus to make those measurements that can be used to predict performance of the adhesive as a high temperature strain gage bonding medium.

The strain transfer into a wire bonded to a structural surface with an adhesive and subjected to a uniform strain is shown in Figure 2. Strain is transferred into the adhesive by shear stresses concentrated at the gage ends and decreases rapidly toward the center of the gage. Only the very end of the adhesive layer is subjected to shear stress, and the tensile strain becomes uniform and equal to that of the substrate before the gage wire is encountered. Strains within the adhesive in the plane of the wire are sketched in Figure 2.

The strain and load in the imbedded wire is zero at the ends but rapidly builds up to a uniform value equal to the uniform strain in the structure. Strain is transferred into the wire by shear stresses between the adhesive and the wire. At the very end of the wire the shear stresses are maximum and the tensile stress/or strain is zero. As the shear stresses transfer load into the wire, these stresses diminish in value. As the strain in the wire approaches the strain in the specimen, the adhesive shear stresses approach zero. The distribution of adhesive shear stresses and tensile load in the wire are also shown in Figure 2.

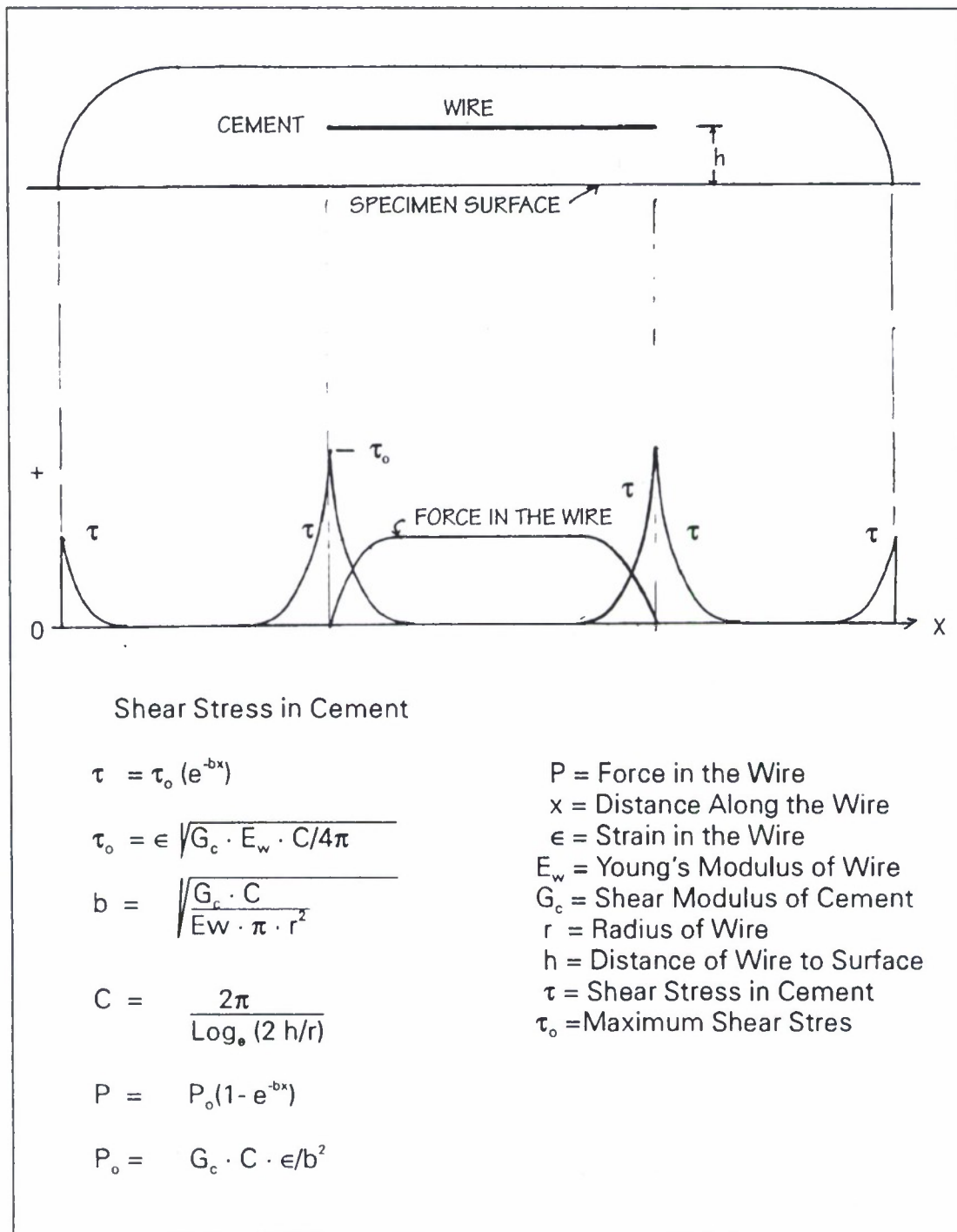


FIGURE 2. Stress Condition for a Wire Bonded to a Strained Surface



The shear stresses in the adhesive are given by the equation: <sup>20</sup>

$$\tau_o = \epsilon \sqrt{G_c \cdot E_w \cdot C / 4\pi} \quad \text{equation 1}$$

where $\tau_o$	=	Maximum shear stress in the adhesive PSI
$\epsilon$	=	Strain in the wire, inches/inch
$G_c$	=	Shear modulus of adhesive, PSI
$G_c$	=	$E_c / 2(1 + \mu)$
$E_c$	=	Young's Modulus of adhesive, PSI
$\mu$	=	Poisson's ratio of adhesive
$E_w$	=	Young's Modulus of wire, PSI
$C$	=	A configuration factor which takes into account precoat thickness and wire size. Assumes infinite thickness
$C$	=	$\frac{2\pi}{\text{Log}_e (2h/r)}$
$h$	=	Precoat thickness, from surface to center of wire, inches
$r$	=	Wire radius, inches

Calculations were made for wires bonded with ceramic cement and Rokide. Since the lead wires are the largest inclusions and require the highest shear stresses in the ceramic bond to bring these leads up to the strain level in the specimen, they are of primary concern. The most common lead wires are:

0.003 inch Diameter Platinum 10% Nickel alloy-annealed  
 0.003 inch Diameter Chromel A (80Ni 20Cr) stress relieved  
 0.005 inch Diameter Chromel P (90Ni 10Cr) annealed  
 0.002 inch Diameter Platinum 10% Nickel annealed.

Young's modulus for the above wires is taken from published values. The tensile stress was calculated from loads measured on the pull tester. The load sensor was calibrated using dead weights and the load measurements at failure were very uniform; therefore, the stress data is accurate.

Young's modulus for Rokide is  $2.8 \times 10^6$  psi from measurements reported by BLH. Young's modulus for ceramic cement was assumed to be  $2.0 \times 10^6$ . This value is an estimated value used at BLH. It was arrived at by comparison of gage factors of free filament strain gages bonded with ceramic cement and the same gages using a Bakelite backing. Bakelite backings had a modulus of  $2.0 \times 10^6$  and produced identical gage factors. Therefore, the  $E_c = 2.0 \times 10^6$  for ceramic cement seems reasonable.

The lap shear stress values for ceramic cement indicates that  $\sigma_s = 390$  psi is a typical value <sup>10</sup>.

Plugging in this value for  $\tau_o$  in equation 1 along with the following:

$$G_c = \frac{E_c}{2(1+\mu)} = \frac{2.0 \times 10^6}{2(1+.3)} = .77 \times 10^6$$

$$r = 0.0015 \text{ inch}$$

$$h = 0.006 \text{ inch}$$

$$E_w = 25 \times 10^6 \text{ (for 0.003 inch Dia Pt 10Ni)}$$

$$\therefore C = \frac{2\pi}{\text{Log}_e (2h/r)} = 3.02$$

$$\tau_o/\epsilon = \sqrt{77 \times 10^6 \times 25 \times 10^6 \times 3.02 / 4\pi} = 2.150 \times 10^6$$

$$\epsilon = \frac{390}{2.150 \times 10^6} = 0.000180 \text{ or } 180\mu \text{ inch}$$

This, then is the maximum strain which can be applied to the gage without fracturing the adhesive in shear, based on a measured lap shear strength of ceramic cement. We know from experience, however, that ceramic bonded strain gages can run endlessly at strains several times this value; say  $> 10^7$  cycles at  $\pm 1000 \mu$  inch strain levels. If we compute shear stresses from our pull test data, we get better values. For a 0.060 inch long bonded length of lead wire and for a 3 mil diameter lead,  $\sigma_s$  can be calculated from the pull test data.

$$P = \sigma_s A_s \quad \text{where} \quad \begin{aligned} P &= \text{Pull force, lbs} \\ A_s &= \pi D \ell \\ \ell &= 0.060 \text{ inch} \\ D &= 0.003 \text{ inch} \end{aligned}$$

The adhesive shear strength computed from the pull test is tabulated in Table 3.

Using the shear stress data from Table 3, the maximum strain which can be applied without fracturing the bond were computed for Rokide and ceramic cement for two precoat thicknesses  $t = 0.003$  inch and  $t = 0.006$  inch, which are typical values. Maximum strain computed for all four lead wires is shown in Table 4.

**Table 3      Physical Properties of Common Lead Wires**

Property	Wire size and alloy			
	0.002 Pt Ni	0.003 Pt Ni	0.003 Cr A	0.005 Cr P
Force, P, lb, required to break wire	0.450	1.013	1.062	1.8995
Shear Area $A_s = \pi DX$ , in <sup>2</sup>	0.000377	0.000565	0.000565	0.009425
Cross Sectional Area of Wire $A_w = \pi r^2$ , in <sup>2</sup>	0.00000314	0.000007	0.000007	0.000020
Stress, Tensile, Wire, $\sigma_{wu} = \frac{P}{A_w}$ , PSI (measured)	143,300	144,700	151,700	94,750
Stress, Tensile, Wire, PSI handbook value	120,000 to 160,000	120,000 to 160,000	120,000 to 180 000	95,000
Stress, Shear, Cement-Wire Bond PSI	> 1790	1790	1880	2010
Modulus of Elasticity of Wire, Ew	25 x 10 <sup>6</sup>	25 x 10 <sup>6</sup>	31 x 10 <sup>6</sup>	30 x 10 <sup>6</sup>

**Table 4      Maximum Allowable Strain,  $\epsilon$**

Cement	Precoat Thickness	Lead Wire			
		Chromel* 0.005 Inch Dia	Chromel* 0.003 Inch Dia	0.003Pt Ni	0.002Pt Ni
Ceramic	0.006	738	800	833	905
Rokide	0.006	628	676	708	772
Rokide	0.003	469	554	578	651

\*Chromel, T.M. Hoskins Mfg. Co., Detroit, Michigan



Note that increased precoat thickness increases allowable maximum strain. This is not, however, a practical way to reduce bond shear stress because it increases the shear stresses between structure and adhesive. Also note, that allowable strains decrease as the wire diameter increases. This is as one would expect intuitively. The allowable stresses also increase when the modulus of the cement is decreased and as the modulus of the wire is decreased. These data show that the pull tests provide much more accurate data than ordinary lap shear tests.

### 3.6 LEAD WIRE PULL-OUT TEST EQUIPMENT

A micropull tester was developed to provide a quantitative measure of bond strength to the strain gage lead wire and to the substrate. Three inch lengths of strain gage lead wire are taped in place over the desired precoat using a tape bar similar to those on free filament strain gages. See Figure 3a. The precoat may be on any desired specimen material (such as carbon composite, Rene' 41 or TZM). The center cutout is exactly 0.060 inch and represents the bonded length of lead on the smallest commercial gages (1/16 inch gage length). The cement, therefore, when used as a strain gage adhesive, must totally transfer strain from the substrate into the lead wire within the 0.060 inch bonded length. The cement bond must have enough strength to yield the wire. We are concerned primarily with the lead wire because it is the largest and stiffest structural component in the strain gage, and represents a significant inclusion in the ceramic adhesive. If the cement does not faithfully transfer surface strains from the specimen into the lead, there will be a movement between the lead and strain sensing wire at the welded connection, which will cause the wire to break.

In operation, usually two wires are bonded along side each other representing an actual pair of leads coming off a strain gage. The lead is bonded in place within the 0.060 inch wide tape cutout and cured according to instructions. Numerous samples can be bonded at the same time to provide statistical data.

The test specimen is then clamped in place on the movable slide. (See Figures 4 through 8.) The free end of the lead wire is inserted into the fine wire clamp at the end of the instrumented cantilever beam. A capacitive sensor measures force on the lead wire. The beam has been precalibrated with dead weights. The output of the sensor (load) is displayed on the Y axis of an X-Y plotter. The time base on the plotter is used to measure movement of the carriage. The test is quick and accurate. Suitable strain gage cements should have sufficient strength to cause the lead wire to break rather than pull out of the cement. The cements should be tested using the same lead wires that will actually be used on the gage. For the tests on this program, 3-mil-diameter platinum-nickel, chromel, and 5-mil chromel are used. This test also measures the bond strength between the cement and the substrate. If this bond is poor, the cement will come off the substrate. The merits of convoluted leads become immediately apparent. Convoluted leads would not pull out of cements from which straight lead wires easily pulled out.

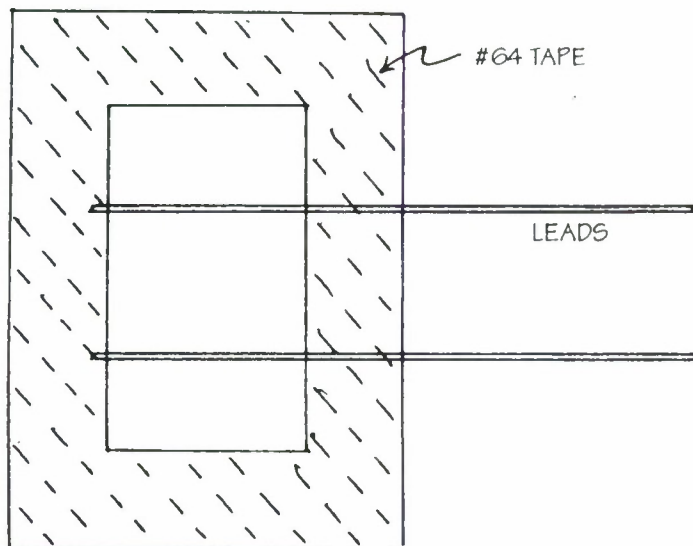


FIGURE 3A.  
Tape Carrier Pull Test  
Specimen Prior to  
Bonding

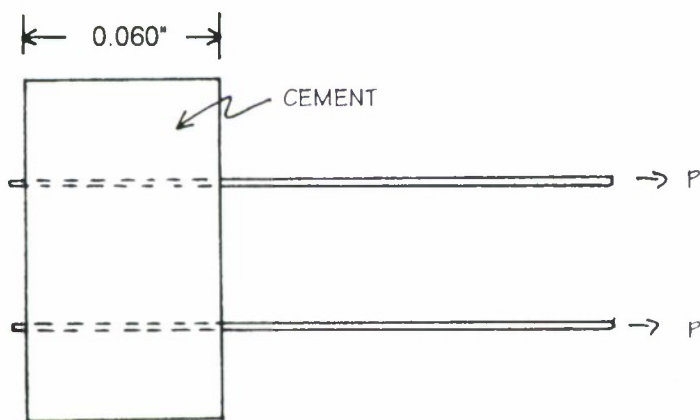


FIGURE 3B.  
Pull Test Specimen  
Ceramic Bonded

FIGURE 3A. Tape Carrier Pull Test Specimen Prior to Bonding  
FIGURE 3B. Pull Test Specimen Ceramic Bonded

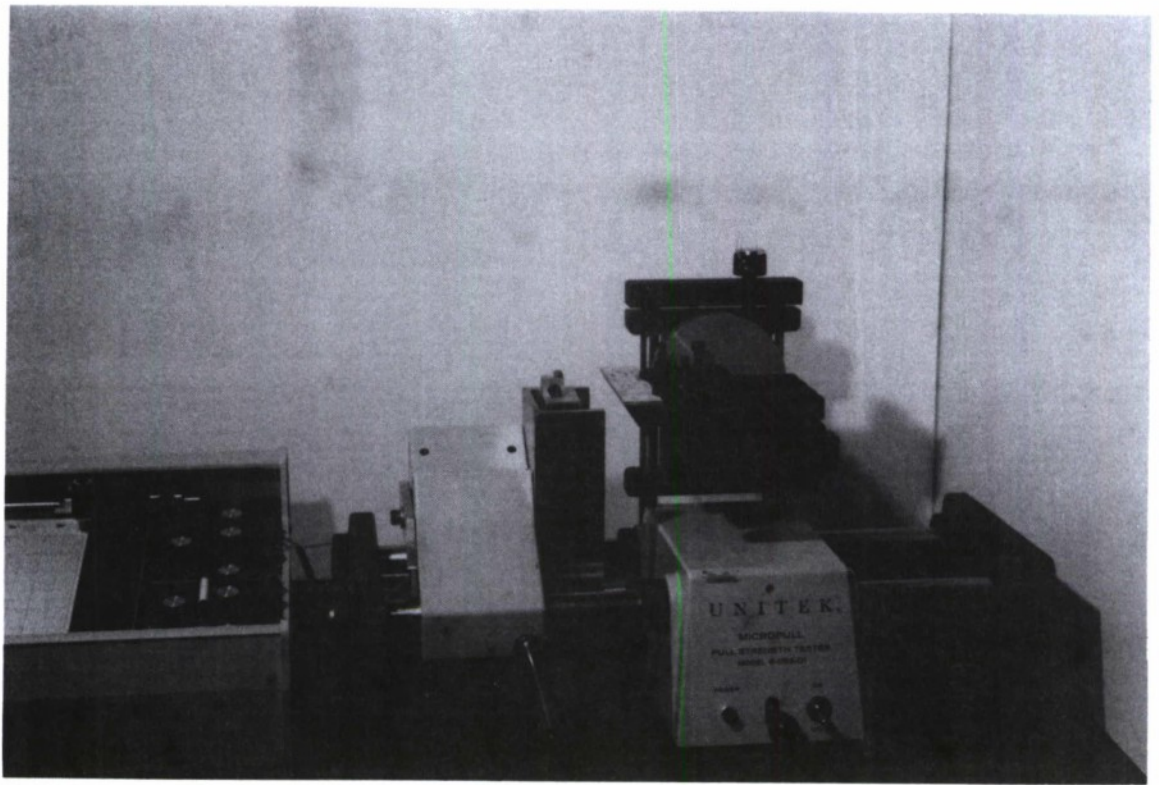


FIGURE 4. Micropull Tester with Recorder

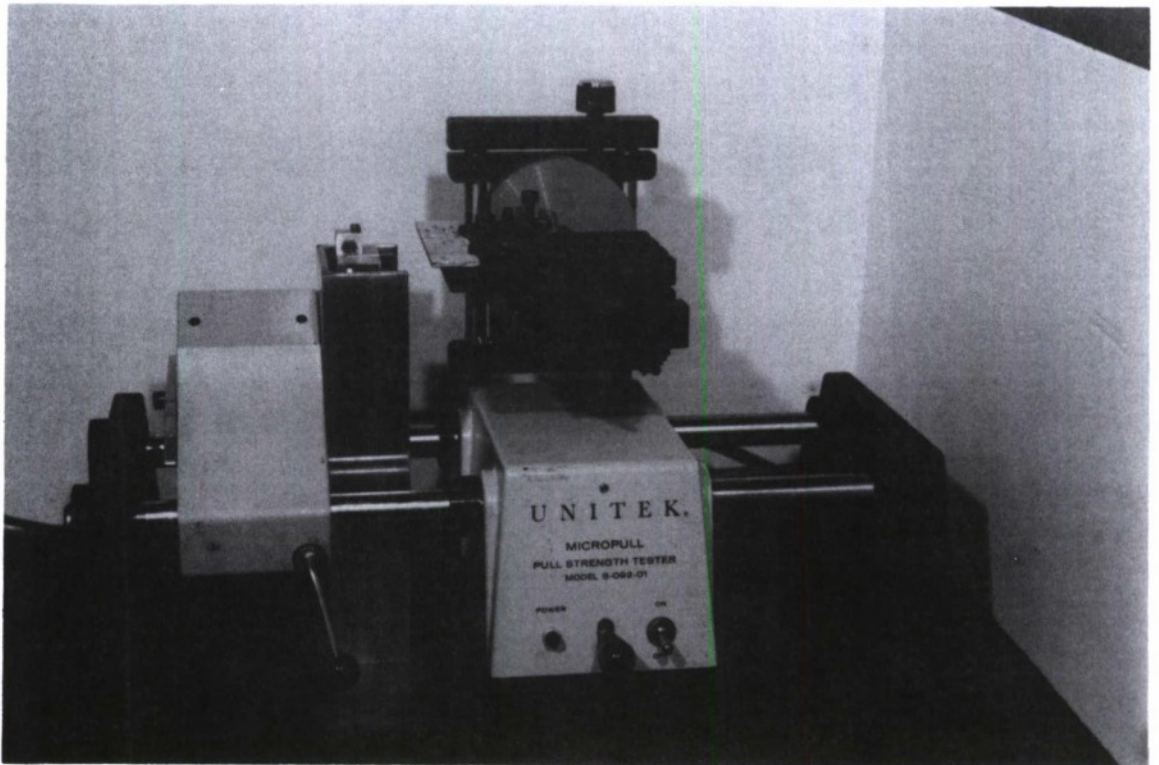


FIGURE 5. Micropull Tester with Capacitive Force Transducer



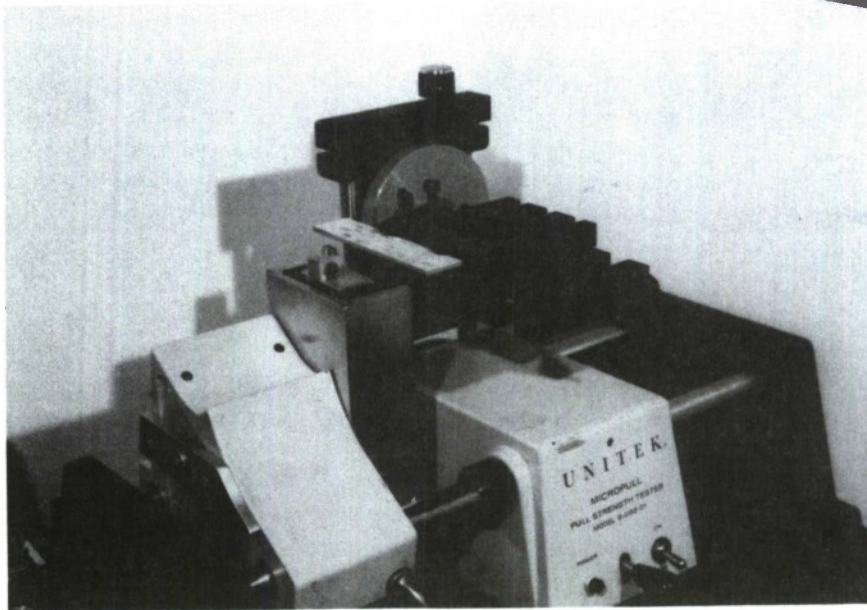


FIGURE 6. Closeup of Micropull Tester

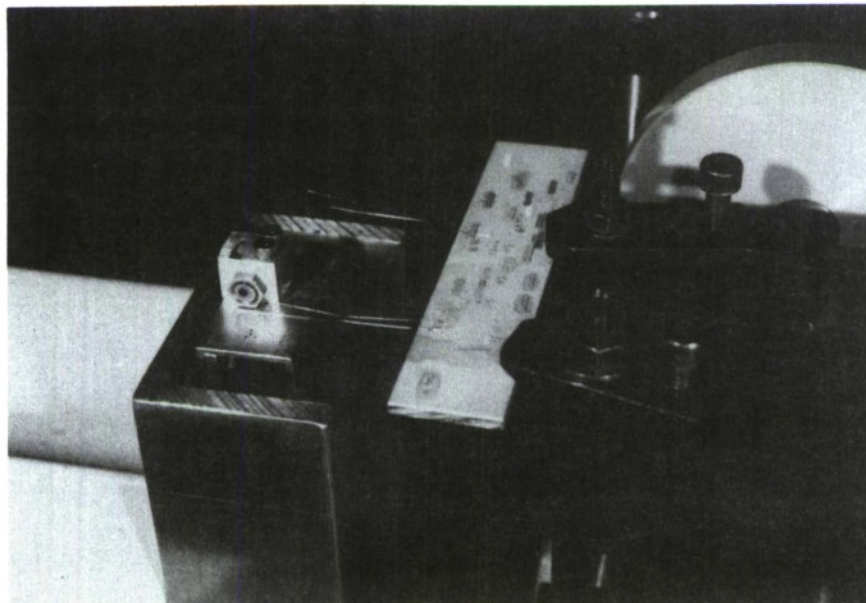


FIGURE 7. Closeup of Specimen Clamped to Table and Wire Clamped at Force Transducer

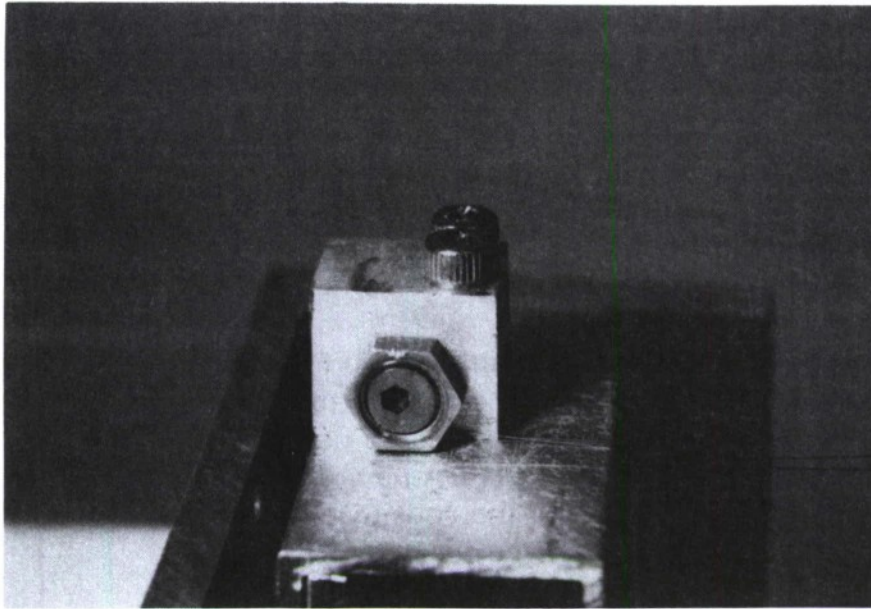


FIGURE 8. Closeup of Cam Operated Fine Wire Clamp

### 3.6.1 Development of the Micropull Tester

A working prototype pull test machine was built. Our intent from the beginning to adapt as much existing hardware as possible to place greater emphasis on developing attachment techniques than on designing a test apparatus. A comprehensive market survey revealed a variety of small testers are available for pull testing components and leads in the semiconductor and electronics industry. A Unitek micropull tester fit our needs almost perfectly and it was purchased. The dial gage load cell was replaced with a capacitive force transducer. The output of the force transducer was displayed on the Y-axis of an X-Y plotter.

A cam type fine wire clamp for gripping the test leads at the transducer was designed and built. See Figure 8 .

The Unitek micropull tester was originally made for pull testing welds. The original specimen clamp was removed and replaced with a Unimat III milling machine table. The test specimen with the prebonded test leads is clamped to the traveling carriage using miniature milling clamps. See Figures 4 through 8. The test specimen is adjusted vertically and horizontally until the lead wire exactly lines up with the fine wire clamp on the force transducer. The lead wire is then clamped in place. To apply load, the machine is turned on and the motor driven carriage applies a tensile load to the wire until fracture occurs.

A miniature resistance heater was designed and built for testing up to 1500°/1600°F. The heater is a miniature hot plate consisting of a nichrome wire element flame spray bonded to a small plate of Inconel. It is installed on the milling machine table using the

same miniature milling clamps as for the room temperature test specimen. A ceramic insulator isolates the milling machine table from the hot plate. The heater is a copy of a design used for heated stages in scanning electron microscopes. Some SEM heated stages reach 2200°F. A heater using platinum elements is usable to 2500°F.

### 3.6.2 Sensing Beam Design

The strain gage based load sensing beam used in Phase I tests utilized an aluminum cantilever beam instrumented with four platinum tungsten strain gages arranged to measure bending. Although this beam could be used directly with the X-Y plotter without amplification, it lacked stiffness. A new design employing differential capacitive sensor (shown in Figure 9) increased the sensitivity substantially, while reducing deflection. This beam design was used for Phase II testing.

### 3.6.3 Bond Specimen Design

#### Bonding problems and sources of error

Some of the long standing problems of attaching free filament resistive strain gages were addressed because of their effect on any bond test evaluation procedure. Some of the problems investigated were:

- A) voids in the cement,
- B) partial bonding,
- C) poor wetting of cement to substrate and wetting of gage grid and leads,
- D) cement shrinkage and the roll of shrinkage on void creation and porosity problems,
- E) crevices caused by improper bonding techniques, and
- D) contamination from various sources as well as the tape carrier itself was looked into in detail. Contamination has a very profound effect on bond strength to specimen as well as to the gage.

The technique and detailed procedures of cement application has a great deal to do with the success of the installation. Cement applied thickly or cured quickly will display voids and porosity. The only difference between porosity and voids is the size of the hole. Methods of mixing and the solids/liquid ratio as well as aggregate size distribution also affect void size and cement strength. The additions of thinner or water to cements will affect bond strength.



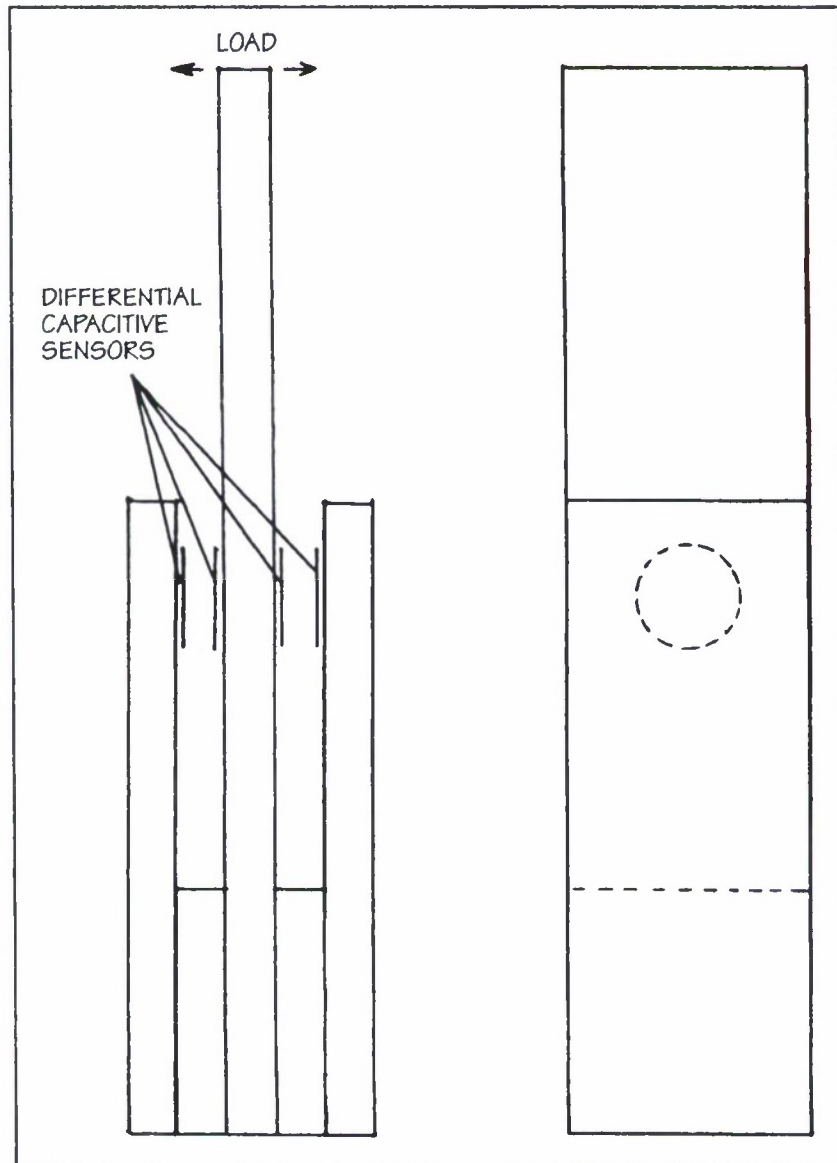


FIGURE 9. Sketch of Capacitive Sensing Beam Design

### 3.6.4 Pull Testing Leads Bonded to Glass Specimens

Test leads were bonded to glass slides to look for defects. It was found that several thin applications of cement produced a more dense ceramic matrix than one thick application. Also, a thick application of cement caused considerable flow of cement along the leads exiting the bond area and partial bonding of these leads for an additional 0.060 inch to 0.080 inch length. The flow of cement occurred in the space between the tape carrier and the slide due to imperfect conformance of the tape to the leads. See Figure 10.

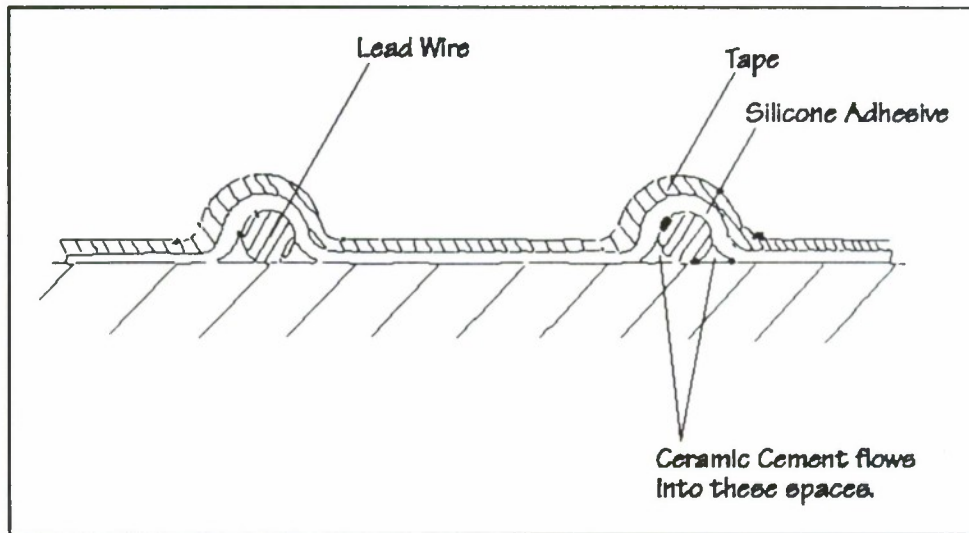


FIGURE 10. Cross Section of Pull-Test Specimen

The cement also bonds to the silicone adhesive under the tape, causing adhesion of a layer of silicone to the cement. This is a major source of silicone contamination. The thickness of the silicone layer adhering to the cement depends on the cure temperature before tape removal - the higher the cure temperature, the greater the thickness of silicone bonded to the ceramic. After a 450°F cure, a lot of the silicone comes off the tape and remains bonded to the part.

### 3.6.5 Observation of Failure Modes

The pull-out process was observed using a stereo zoom microscope mounted on the pull tester as shown in Figures 11 and 12. Pull-out test samples were bonded to glass microscope slides, and the failure process could be clearly viewed as the wire pull-out progressed. The motorized carriage of the pull tests moved too fast for careful observation, so a screw drive was attached to apply load manually. Bond failure occurred during yielding and necking down of the lead at the cement bond interface. The failure was not a shear failure between the lead and the cement but a tensile failure which occurred as the lead necked down, decreasing the lead diameter due to Poisson's strain and pulling away from the cement in a tensile stress condition. As load is





continued, the crack at the interface propagates by progressive necking of the lead wire followed by a tensile separation between the wire and the cement matrix. As the wire necks down and debonds, load transfers to the remaining bonded wire. Ordinary glass slides were used to observe failures during pull-out using standard cement cure schedules, and quartz glass slides were used to observe pull-out for cements cured at higher temperatures.

### 3.6.6 Room Temperature Pull Tests

A precoat of selected adhesive is applied to a specimen and cured (any convenient size specimen could be used). Two leads are placed on the test bar and held in place with a tape carrier during bonding (see Figure 3). The carrier is similar to that used on free filament strain gages with a precision 0.060 inch wide cut out in the cement bonding area. The tape carrier is double thickness 3M no. 64 tape. The test cement is applied and cured using standard procedures. The tape is removed and the specimen mounted in the micro-pull tester and pull tested. See Figures 13, 14, and 15 showing specimens after pull testing.

The average pull strengths for Rokide bonded specimens are shown in Table 3. Rokide was found to produce the strongest bond. In no case did the wire pull out of the Rokide. All failures were wire failures. Some of the ceramic cements were capable of breaking the wire as well as displaying remarkable uniformity in the force required to pull the wire out of the cement. This suggests the mode of failure is a necking down of the stretching wire, thus debonding due to tensile stress rather than shear. The necking down of the wire would propagate into the cement until the failure traversed the 0.060 inch bonded length.

Why did the necking down not happen with Rokide? One possible explanation is that shrinkage of the Rokide during cooldown from 3000°F produced a very tight encapsulation of the wire producing initial compressive stresses.

#### 3.6.6.1 Pull Tests on Ceramic Cements and Flame Sprayed Coatings

The room temperature pull tests of the following materials were completed during this program:

OS-82-01	(Russian)	GA-100	(U.S.)
P12-9	(Chinese)	CR-760	(German)
Brimor	(British)	WC-9	(U.S.)
Bean H	(U.S.)	Rokide Hitec HT	(U.S.)
PBX	(U.S.)	FA Powder	(U.S.)
Yellow Cerro	(U.S.)		

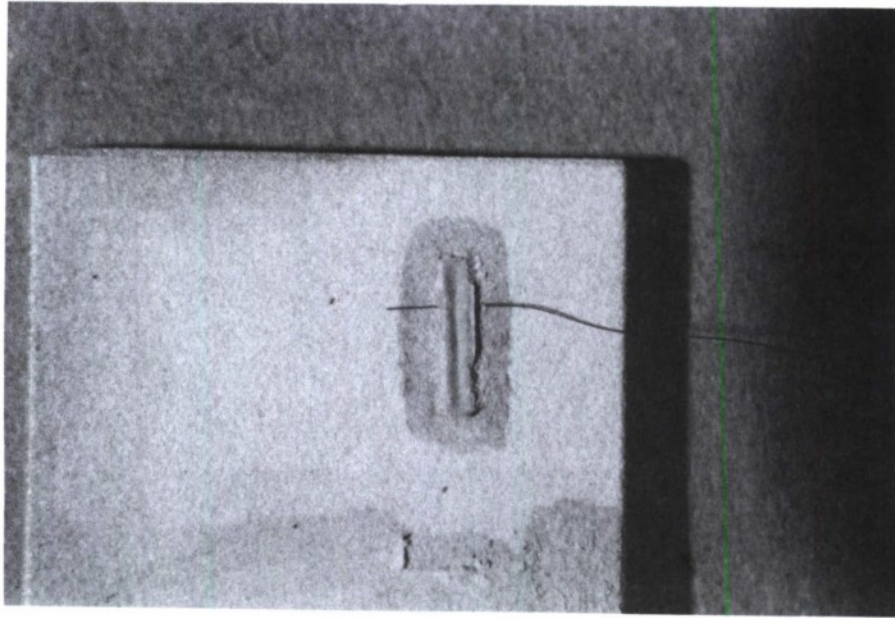


FIGURE 13. Closeup of a Lead Wire Bonded to the Test Specimen. The lead wire broke rather than pull out of the cement. The second wire (not shown) pulled out of the cement.

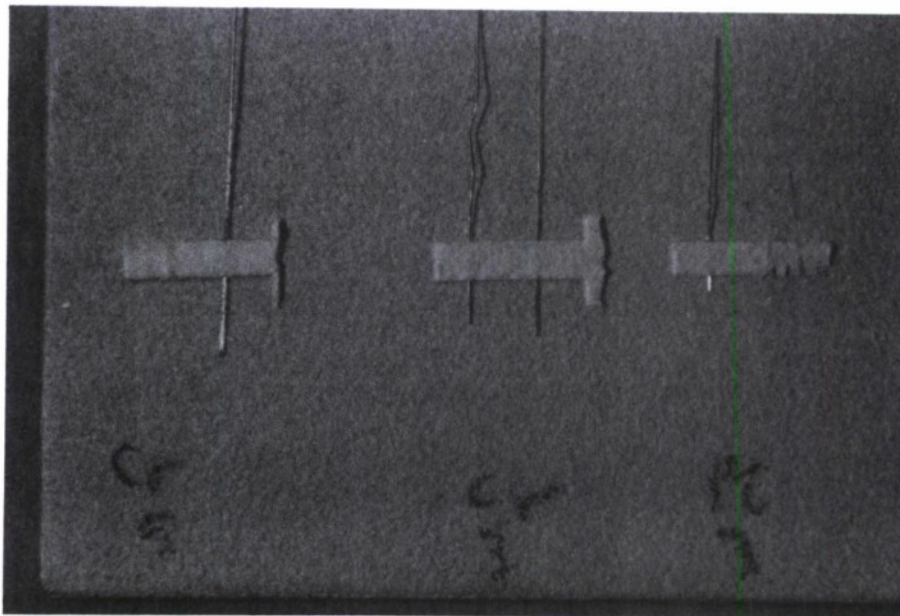


FIGURE 14. Rokide Bonded Leads, Left, 0.005 Inch Dia. Chromel, Center 0.003 Inch Dia. Chromel, Right .003 Inch PtNi

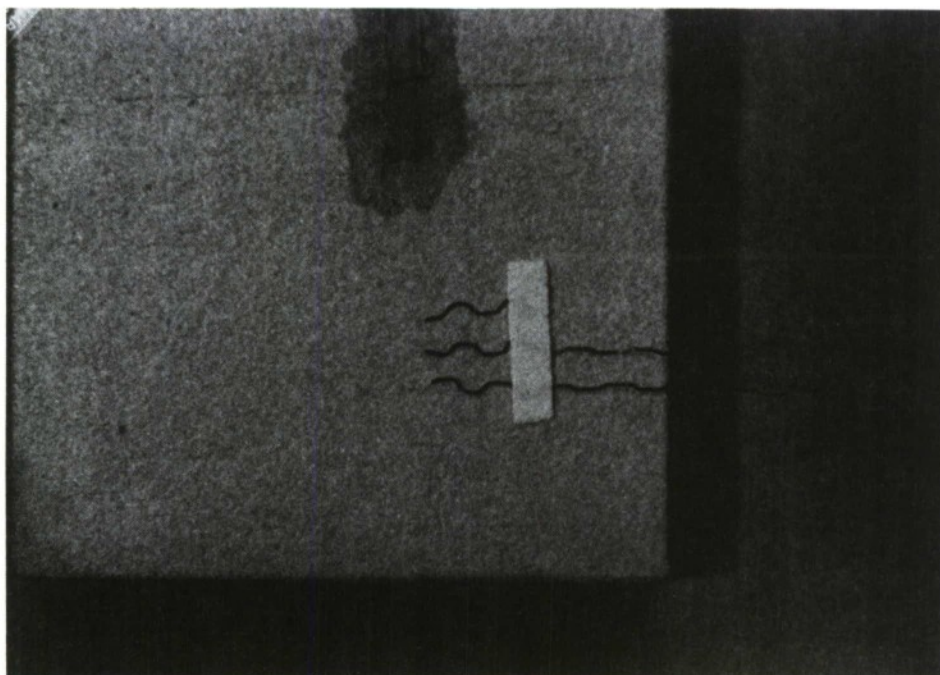


FIGURE 15. Powder Flame Sprayed FA Powder and 0.002 Inch Dia. Convolute and Flattened PtNi leads. All leads broke rather than pull out of the coating.



Tests were conducted on 1 inch x 4 inches Rene' 41, 1 inch x 3 inches clear glass specimens, 1 ½ inches x 1 inch quartz specimen, and ½ inch x 4 inches uncoated carbon composite specimens. All of the materials tested exhibited sufficient strength, using 0.003 inch Dia Pt Ni leads, to cause the lead wire to yield. The strongest bond, by far, was produced by Rokide flame spray methods, which produced ultimate breakage of the wire in every test.

#### 3.6.6.2 Effect of Cement Age on Bond Strength.

Pull tests were run on 0.003 inch Dia. platinum nickel lead wires bonded with fresh cement and 1-year-old cement. Both Yellow Cerro and Chinese P12-9 cement were tested. The fresh P12-9 cement was 20% to 30% stronger than 1-year-old cement. The same results were found for the Yellow Cerro cement. Although both cements had adequate strength at 1 year to transmit strain, fresh cement is better and stronger. The X-Y plots of one of these tests are shown in Figure 16.

#### 3.6.6.3 Effect of Cure Temperature on Bond Strength.

Yellow Cerro cement was pull tested after various cure temperatures to profile the strength vs cure temperature curve. The data are plotted in Figure 17. It is important to identify the minimum cure temperature required before removing carrier tapes. The 0.003 inch Pt Ni leads were bonded to Rene' 41 test specimen and cured at 200°F, 300°F, 400°F, and 620°F. At 200°F cure, the cement had no measurable strength. At 300°F cure, the cement was nearly at full strength. The 400°F cure had results similar to the 300°F cure. The 620°F cure was the strongest. Fresh Yellow Cerro cured at 300°F has more strength than fully cured Yellow Cerro 1 year old. This information indicates that the carrier tape can be removed from the gage grid after a 300°F cure. This minimizes silicone contamination which occurs, to a large extent, after the 400°F cure.

#### 3.6.6.4 Effect of High Temperature Cure on Cement Strength

The Russian orthosilicate cement OS-82-01 was tested comparing a 600°F cure with a 1025°F cure. The pull test results are plotted in Figure 18. Note that the 600°F cure shows adequate strength while the 1025°F cure, the strength has substantially decreased.

W.B. = WIRE BROKE  
P.O. = WIRE PULLED OUT OF CEMENT

0

CAL

TIME

YELLOW CERRO (OLD)  
670°F CURE

CHINESE P12-9 (FRESH)  
LOT 4892  
620°F CURE

CHINESE P12-9 (OLD)  
LOT 4891  
620°F CURE

30

HITEC PRODUCTS INC.  
 AYER, MA 01432  
 WIRE PULL TESTS  
 0.003" DIA. PtNi  
 UNITEK PULL TESTER  
 500 mv/in; 1 LB = 1.OV.  
 4-10-92 SPW

YELLOW CERRO CEMENT LOT 32392  
 RENE' 41 TEST SPECIMEN

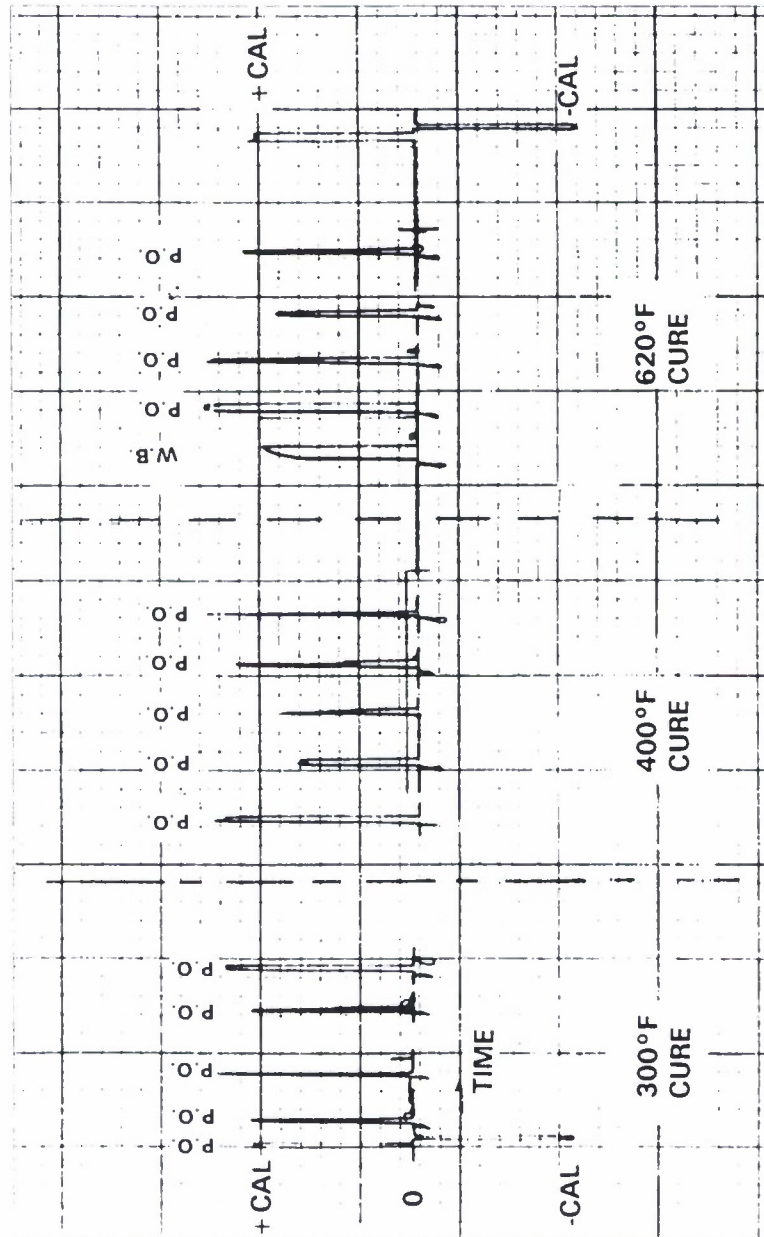


FIGURE 17. Cure Temperature vs. Strength



HITEC PRODUCTS INC.  
 AYER, MA 01432  
 WIRE PULL TESTS  
 0.003" DIA. Pt 10Ni  
 UNITEK PULL TESTER  
 500 mv/in; 1 LB = 1.OV.  
 3-31-92 SPW

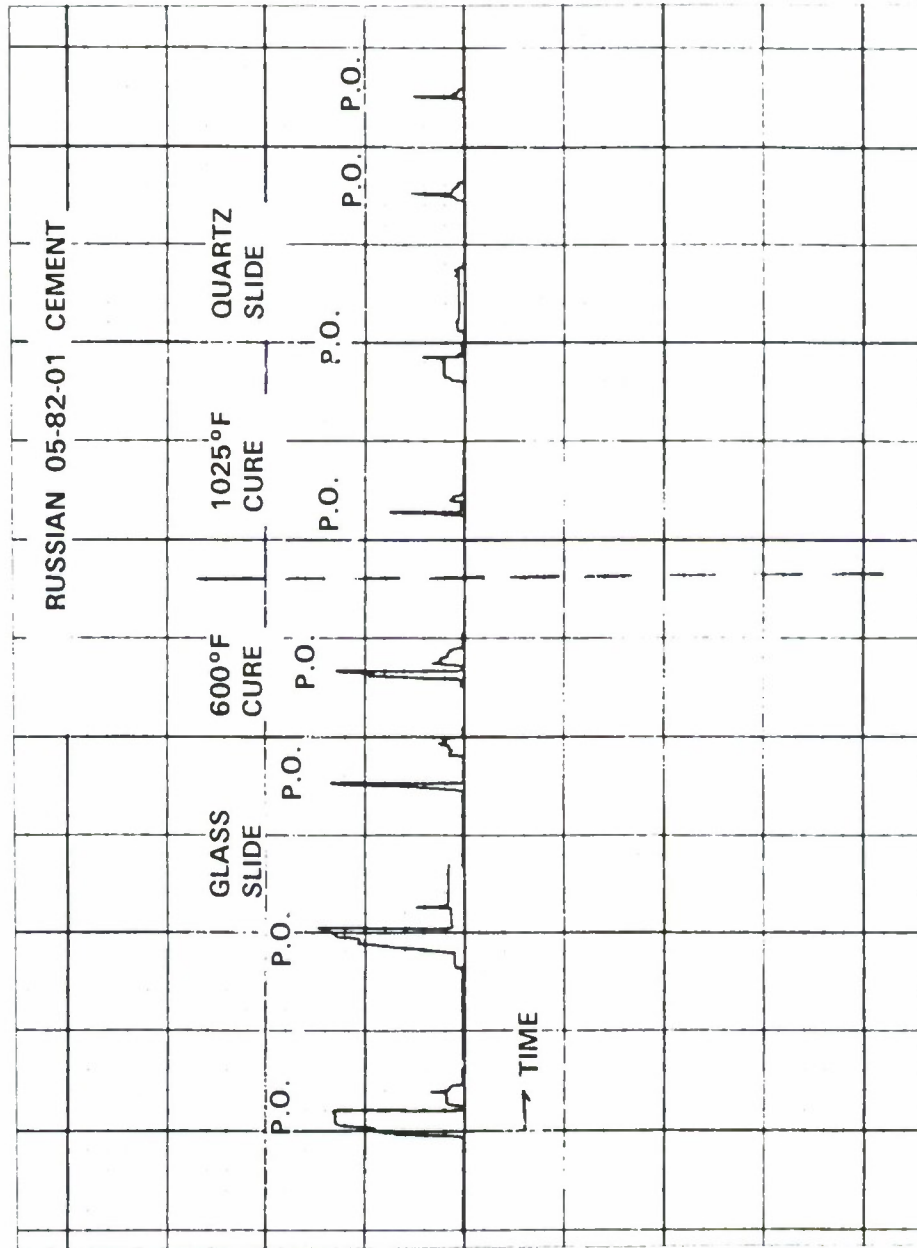


FIGURE 18. Pull Strength of Russian Cement

### 3.7 THEORY OF ADHESION

#### 3.7.1 High Temperature Bonding

High temperature adhesives used for strain gage bonding are basically two types:

- A) ceramic cements consisting of a ceramic aggregate mixture suspended in a liquid medium and,
- B) a melted ceramic such as flame sprayed aluminum oxide materials used to bond to the test specimen and to embed the strain gage in a thin layer of coating material.

In order for an adhesive to stick to a test material, it must be able to come in full contact with the material. The first test of an adhesive is in its ability to wet and spread on the test material. If the adhesive will not "wet" it will not produce a good bond. Surface preparation therefore becomes an important part of the bonding procedure. Surface preparation involves removal of contaminants and films, oxides, scale, everything which could prevent the test material from coming in full contact with the adhesive.

According to the adsorption theory of adhesion, which has been the most widely accepted theory<sup>22</sup>, any two materials will adhere to each other if they are brought close enough to each other. The key to strong adhesive joints is extensive and intimate intermolecular contact. However, the intermolecular force diminishes as the seventh power of the distance. The two materials must therefore approach within a few angstroms of each other if they are to adhere. The only way to achieve this contact is for one of the materials to be a liquid. For the best joint strength, the liquid must be more than fluid, it must spread. An adhesive that will not spread will not flow into the surface crevices very easily, but will simply bridge across the surface mountains and will therefore form a weak joint. If the molecules of the liquid stick better to the solid than they do to themselves, the liquid will spread.

Another rule in the adsorption theory states that if one material does not wet another, if the roles are reversed, wetting will occur. This is born out by the fact that, in general, metals will not wet ceramics. This is evident in ceramic crucibles used for melting metals and in sand or ceramic molds used in metal casting. The molten metals do not wet and hence do not stick. However, if the roles are reversed and the ceramic is melted, it will wet and stick to the solid metal. This is precisely the case for ceramics flame sprayed onto metals.

Microscopic examination during flame spraying of fine wire metal sensors reveals that wetting of the sensor by the flame sprayed ceramic does occur. Often debonding occurs during cool down, probably due to thermal stresses caused by the difference in thermal expansion between the ceramic and the metal wire.

### 3.7.2 Ceramic Cements

Ceramic cements are aggregate mixtures suspended in a liquid medium consisting of phosphoric acid or mono-aluminum phosphate, which accomplishes several tasks:

- A) The liquid wets the specimen and brings the aggregate mixture into intimate contact with the specimen material.
- B) The liquid dissolves or displaces any contaminants on the surface as well as absorbed moisture or gases.
- C) Some liquids, by design, remove some of the oxides on the specimen surface and may deposit an adherent film which will readily accept the bonding medium.
- D) The liquid readily wets the strain gage grid and leads and etches or dissolves minor contaminants and absorbed gases.
- E) The liquid medium keeps the ceramic aggregate in suspension during application and distributes it appropriately about the free filament.

The liquid medium, having accomplished the above, then evaporates leaving the binder and ceramic aggregate to bond to the test specimen and gage. As the liquid medium evaporates, the aggregate settles and comes together slowly and begins to lock into place. Too rapid fluid evaporation can cause problems. The top layer of the cement may become hard and form a skin which prevents settling into the more dense fluid. If this happens, the thin skin forms sort of a roof or egg shell surface which will not settle, but creates voids in the cement matrix. This phenomenon frequently happens in ortho silicate cements under rapid drying conditions. It can be avoided by increasing the room temperature drying time or by thinner applications of cement. Thick coatings have problems with porosity and voids. It is better to apply more thinner coatings than less thicker ones.

#### 3.7.3.1 Bonding Gages to Glass

To study the problems associated with ceramic cements, free filament strain gages were bonded to glass microscope slides using Yellow Cerro cement. HFP-12-250-XPW strain gages were attached using standard instructions and cure schedules and then observed from the underside for defects under a microscope at magnifications from 7x to 70x. With the gages bonded to glass, the interior of the strain gage installation could be easily examined. One of the desirable features of Yellow Cerro is that it contains no pigments, and is therefore semitransparent when wet. Air bubbles or incomplete coverage often can be observed during application and dealt with while the cement is still fluid. Examination of the slides revealed a surprising number of defects in the cement even



though from the topside the installations looked near perfect. Bubbles, voids, crevices and imbedded silicone adhesive (from the tape carrier) were found on some installations which looked good from the top side.

Strain gages were also installed on glass slides using Rokide. See Figures 19 and 20. In general, Rokide installations had less voids than the ceramic cement. Crevices and silicone contamination are also greatly reduced. Some of the improvements in attachment techniques developed during this program are responsible for the nearly defect-free Rokide installations on slides. The detailed procedures used are documented in Appendix B.

### 3.7.3.2 "Lock-up" Cure Temperature of Ceramic Cements

In order to minimize silicone contamination from temporary carrier tape adhesive, it is important to find the minimum cure temperature for ceramic cements. The silicone adhesive in the tape begins to cure at 400°F, with full cure developing at 500°F. If the ceramic cement can be cured below 400°F, say at 350°F, then silicone contamination can be minimized.

The liquid portion of six cements was placed on a microscope slide, cured at various temperatures, then examined under the microscope and checked for hardness. The liquid was allowed to remain at room temperature for extended periods of time with relative humidity noted, then reexamined for hardness. The cements tested were Yellow Cerro, WC-9, Hitec 701, Chinese P12-9, Yellow Cerro Mod 1, and Bean H.

The results indicate all cements required at least a 300°F cure in order to stay hard when exposed to extended periods (days) at room temperature and humidity of 38 - 42%. All cements when cured at 230°F were hard initially, but turned very soft after 6 days at room temperature and R.H. of 38 - 42%. Therefore, if the carrier tape is removed after the 200/225°F bake, it must be followed up with a 350/400°F bake to permanently harden the cement prior to any extended stay at room temperature. These tests confirm the little known fact that the cement hardening process is reversible when cured below 300°F. A 300°F or higher cure is required to permanently harden the cement.

These tests also demonstrated that the liquid bubble in the drop of liquid cured from the outside in. The outer surface in contact with the air hardened and formed a very thin shell, through which liquid evaporated leaving an inner void. If cured rapidly, the hard outer shell crazed, or cracked. The hardest bond surface is the thin contact area between the bubble and the glass slide.

This bubble formation has also been observed in cements which have excessive amounts of liquid component. The excess liquid forms small bubbles or flakes on the surface of the ceramic giving it a jaundiced appearance. The effect of excess liquid component on bond strength was planned for study but time did not permit its completion.

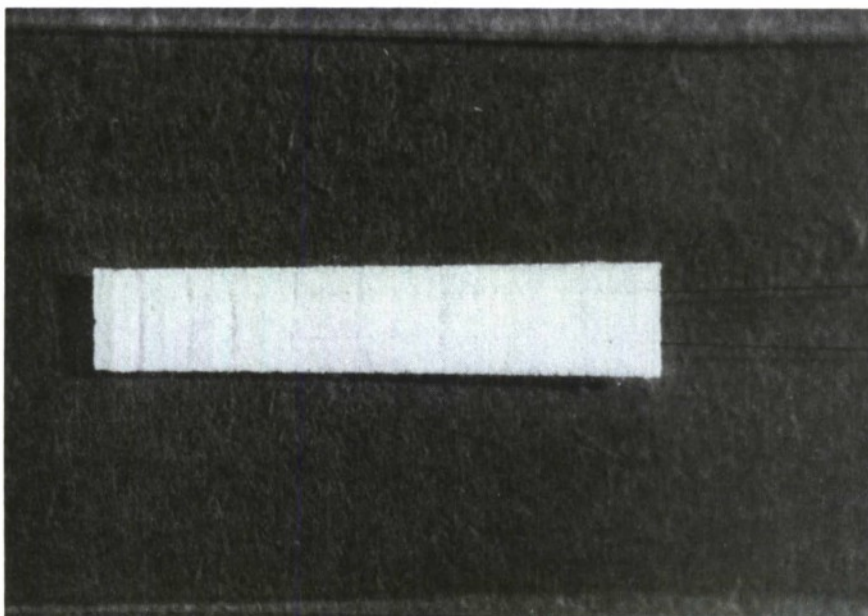


FIGURE 19. Rokide Bonded Strain Gage Attached to Glass Microscope Slide. Viewed from top.

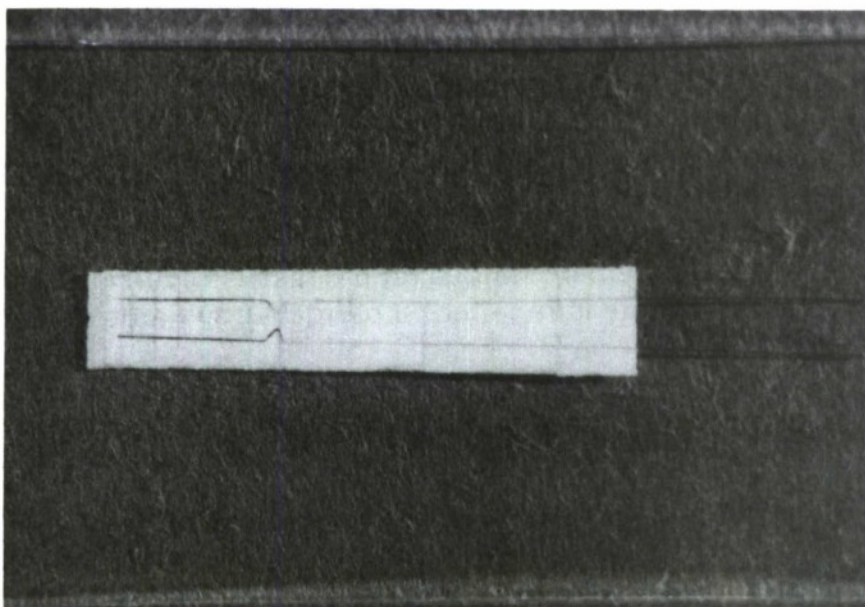


FIGURE 20. Rokide Bonded Strain Gage Attached to Glass Microscope slide. Viewed from bottom.

### 3.7.4 Silicone Contamination Studies

Nearly all of the high temperature attachment techniques use tapes in one form or another containing silicone adhesives and silicone contamination has always been, and still is, a major problem. This problem was studied and development of procedures which eliminate, or at least minimize, silicone contamination were addressed. The principal causes of silicone contamination are:

- A) low quality tape, and
- B) improper tape handling procedures.

In addition, we tried to carefully identify which procedures cause silicone contamination and to develop application procedures which eliminate or minimize the problem. Quartz glass microscope slides were used in this evaluation because they can withstand high cure temperatures, and are optically transparent so that cement bonds can be observed from the backside under a microscope.

#### 3.7.4.1 Tape Quality Control

Tapes used for fabricating free filament strain gages are routinely quality controlled by testing tape samples on glass slides. Tape samples are bonded to the glass and evaluated after various time periods. The evaluation consists of cutting the tape into very narrow strips (0.030 inch wide) and peeling these strips from the glass. The silicone adhesive bond to the tape is observed and the glass is observed for adhesive residue. If residue occurs, or if the adhesive debonds from the tape, the tape lot is rejected. Tape which passes initial inspection is set aside or used for lead wire attachment bonding. After a month the peel test is repeated. If the adhesive does not leave a residue on the glass and does not debond from the tape, the tape is then used for making test gages, some of which are bonded using ceramic cement and some bonded using Rokide. If there are no problems with either the cement or the Rokide applications, the tape is then labeled "acceptable for making gages." There are a large variety of silicone-fiberglass adhesive tapes on the market, but very few are found "acceptable for making strain gages."

#### 3.7.4.2 Cure Temperature vs. Tape Residue

Strips of tape 0.1 inch x 0.6 inch were bonded to a silica (quartz) microscope slide, heat cured at various temperatures and evaluated for silicone contamination. Ceramic cement wettability in the tape area after tape removal was used as the criteria to determine whether residue was present. Residue was found at each tape location after tape removal. Even after brief room temperature tape contact, the taped area wettability was very poor until mechanical removal of the residue, by rubbing with a gauze pad soaked in MEK, was accomplished. As the cure temperature increased, removal of the residue became more difficult. At 400°F, a pronounced increase in adhesive bond strength to



the glass substrate occurred, and it was very difficult to remove the tape with the adhesive intact. Cleaning was also very difficult before ceramic cement wetting would occur. After a 500°F cure, about 50% of the silicone adhesive remained on the glass after tape removal. Ceramic cement would not wet after hard cleaning.

After a 600°F cure the results were about the same as the 500°F cure. After a 700°F cure, about 50% of the silicone adhesive disappeared. The fiberglass lay loose on top of the remaining adhesive. Ceramic cement would not wet the specimen anywhere near the adhesive, (even 1 inch away) as though the silicone had vaporized and contaminated the whole area nearby. The adhesive was flaky but still well bonded. After 24 hours at 700°F, ceramic cement still would not wet the contaminated area.

We found that 2 hours at 800°F were required to effectively heat clean the silicone contamination. Because of the gradients in most furnaces between front to back and vertical, a somewhat higher temperature setting should be made to be sure the specimen has been exposed to at least 2 hours at 800°F for effective heat cleaning. At this temperature, the silicone adhesive is effectively converted to silica powder, which will either blow away or readily mix with the ceramic cement.

We found, in separate experiments earlier, that silicone contamination of ceramic cement or Rokide precoats can take place at room temperature if the adhesive is in contact with the precoat for a long period of time (several days). See Figure B-18 in Appendix B.

The results of these experiments therefore suggest a set of rules which should be followed by strain gage installers in order to minimize or eliminate silicone contamination and its potentially disastrous results.

#### 3.7.4.3 Silicone Removal

Three methods of silicone removal used in the industry are:

1. Application of Methyl Ethyl Ketone (MEK) to swell the silicone and mechanically remove same using a stiff brush, tweezers or pick.
2. Apply acid based ceramic cement thinner to etch the silicone away. This is followed by a thorough washing procedure to remove all the acid. This procedure does not work. The acid passivates the silicone and wetting occurs but the silicone is still present, disaster results.
3. Bake the contaminated specimen at 800°F for 2 or more hours followed by a dry brush removal of residue.

Only one procedure worked 100% of the time: the 800°F bake for 2 hours (700°F for 6 hours did not do the job but seemed to further contaminate the whole area near the tape). Cleaning with MEK also works if the cleaning is extremely thorough.

#### 3.7.4.4 Preventing Silicone Contamination During Gage Bonding.

##### 3.7.4.4.1 General Rules

- A. Specimen Cleaning: After solvent cleaning and prior to grit blasting or nickel aluminide application, the specimen should be heat cleaned in an oven for 2 hours at 800°F. Solvents alone cannot remove silicone.
- B. Keep virgin surfaces virgin: This means that surfaces once heat cleaned, must not be touched by silicone adhesives.
- C. Lead wire paths must also remain virgin: When masking the lead area while installing the strain gage, mask the lead area using thin sheet aluminum taped or clamped in place.
- D. Keep the heat cleaned specimen clean: Precoats of ceramic cement or Rokide should be applied promptly followed by gage and lead wire installation. If any part of the process cannot be done promptly, the grit blasted or precoated specimen should be protected from the atmosphere by placing the specimen in a sealable plastic bag or suitable container. Machine shop atmospheres contain vaporized silicone cutting oils or other cutting oils which are readily absorbed into Rokide or ceramic cements.

##### 3.7.4.4.2 Ceramic Cement Installation

- A. Keep the tape adhesive contact time with the precoat at a minimum. Do not apply a gage unless the tape carrier can be removed before going home for the day or before going to lunch.
- B. When applying ceramic cement, do not touch the tape or adhesive with the ceramic cement. Use a tiny brush and stay well clear of the tape.
- C. Keep laboratory humidity low - never exceeding 47% but preferably below 35% R.H. This will minimize air drying time. Keeping the tack coat as thin as possible also minimizes drying time.
- D. Heat cure the tack coat of cement for 20 minutes at 200/220°F followed by 5 minutes at 300°F to 325°F. Fan cool.

- E. Promptly remove tape carrier completely. At this point in the process there are two alternatives: the preferred procedure, and the alternate procedure.
- F. Preferred Procedure: Remove all tape from the specimen and bake at 300°F to 350°F for 1/2 to 1 hour, 650°F for 1 hour, and 800°F minimum for 2 hours. Furnace cool .

Alternate Procedure (When it is impossible to remove all tape from the specimen and heat to 800°F): Using a clean artists brush and MEK, clean the gage grid area by brushing (in one direction only) repeatedly, at least 20 times. This procedure mechanically removes any silicone residue out of the gage grid area. The MEK causes the silicone to swell and the brush mechanically moves the silicone out of the bonding area. This is a long, hard, tedious process, but adequate cleaning can be achieved as long as the adhesive has not been exposed to 400°F temperature, after which only heat cleaning will be adequate. The surface is clean only if the ceramic cement readily wets the precoat on the first try. If the ceramic cement must be coaxed into wetting by persistent brushing action, this action will passivate the silicone and the ceramic cement will bond to the silicone, resulting in future void formation or delamination.

- G. After the 800°F bake, or alternate procedure, apply cover coat to gage grid and leads. Keep the cover coat as thin as possible, but cover everything with a nice uniform blended coat. It is best to cover everything with one coat only, because subsequent touch up coatings are very difficult to accomplish. The substantial ceramic mass quickly absorbs all the liquid component from the touch-up brush resulting in a dry cement which is very difficult to spread where wanted, and usually results in too thick a patch. The final coat should be air dried for a long time, 1 to 6 hours or more, then cured by slow heating to 200°F for 1 hour, 400°F for 1 hour and 600°F for 1 hour. Slow heating and a longer time at temperature, especially at the lower temperatures is beneficial.

#### 3.7.4.4.3 Flame Spray Installations

- A. Keep the tape adhesive contact time with the precoat at a minimum (as in 3.7.4.4.2a above).
- B. Do not move the gage around on the precoat by repeatedly lifting and placing. Hold the gage with tweezers exactly over the desired location and lower the gage into place onto the precoat. Moving the gage after placement only contaminates more virgin precoat area as well as reducing the bond strength of the tape.
- C. Route the leads and tape them in place using strips of double thickness no. 64 tape 0.050 inch wide. Press directly down on all the tape bars using blunt



tweezers to be sure all the grid, lead and tape is in intimate contact with the precoat.

- D. Place another strip of double thickness no. 64 tape 0.010 inch wider than the gage tape bar directly over each gage tape bar. Center the tape so it overhangs the gage tape bar by about 0.005 inch on each side.  
(See Figure 21.)

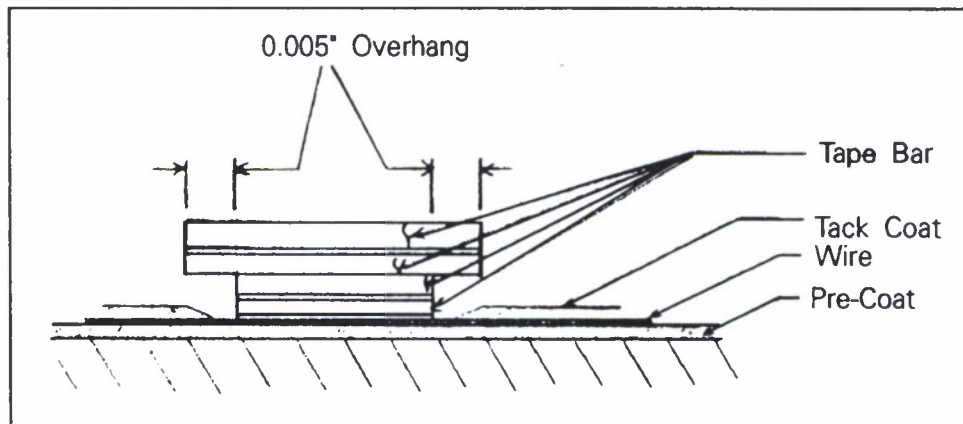


Figure 21. Cross Section of Free Filament Gage Prepared for Flame Spray Installation

This additional double thickness tape bar accomplishes two objectives:

- I) the additional tape thickness keeps heat away from the adhesive in contact with the precoat and therefore reduces silicone contamination, and
- II) the overhang keeps the flame sprayed particles away from the adhesive preventing adhesive entrapment. It also provides a nice tapering off of the tack coat requiring no dressing off of sharp corners after tape removal. This tapering off of the tack coat allows gradual and uniform blending with the cover coat, eliminating crevices or other stress concentrations.

- E. Box in the gage area to be coated using single thickness no. 64 tape.
- F. Apply clean, filtered, cooling air across the gage grid using an air nozzle or a vortex type air cooler. The vortex air cooler is preferred because it will keep the gage installation below ambient temperature during flame spraying. One major source of silicone contamination is overheating the gage tape bars from

the flame and particle impingement. Fast passes with long rest time between passes along with air cooling are necessary to keep the tape adhesive cool. It is important that the cooling air be filtered to remove all contaminants, especially silicone oils used in the compressor.

- G. The technician can check gage grid temperatures during spraying by installing a free filament resistance thermometer wired to an X-Y plotter. The thermometer is used to record grid temperature vs. time during spraying. At no time should the grid temperature exceed 400°F.
- H. All tapes should be removed immediately after spraying and be remasked for the final coat. The installation is boxed in using single thickness no. 64 tape extending 1/32 to 1/16 inch outside of the first boxed tape location. This permits a gradual tapering or blending of the perimeter of the gage matrix. After the cover coat, further blending is done using an oil free, white, aluminum oxide abrasive stone about 60 grit. The tape blend reduces the stress concentration at the ceramic matrix - specimen interface.
- I. Following the above procedure reduces thermal stress in the coating and minimizes silicone contamination. The 3000°F particles of alumina readily convert any minute amounts of silicone into aluminum silicates that are not detrimental to the installation. In addition, the quad tape thickness with shaded area promotes blending of the tack and cover coats and eliminates crevices which cause poor gage performance.
- J. Protection during lead splicing: If the leads are to be soldered or brazed, place a piece of teflon film over the Rokide or ceramic cement and tape in place using no. 64 tape, covering the exposed Rokide. As the joint is made, flux vaporizes and contaminates everything in sight. Clean the joint of flux residue and thoroughly clean the area prior to removing the protective tape.
- K. After joints and lead splices are completed, the installation should be placed in a sealed bag or cabinet to prevent atmospheric absorption of contaminants. A piece of no. 64 tape lightly bonded over the installation will protect the installation from absorbing contaminants or moisture. A double thickness of no. 64 tape will waterproof a gage installation and has demonstrated resistance to water penetration while submerged in water for up to 1 week. Unless one is going to bond something to the finished installation, silicone contamination at this point is of no consequence.

### 3.7.5 Wire Temperature During Flame Spraying

Heating of the gage wire during flame spraying comes from two sources: 1) heat from the molten ceramic particle transferred into the wire as the particle impacts the gage installation and 2) heat from the turbulent air and burning gasses as the coating pass is made over the gage. The heat from the molten particle can generate very high temperature, which according to Hudson<sup>23</sup> can approach the melting point of the wire. This high temperature is extremely localized, primarily confined to the dimensional boundary of the molten particle. Most of the heat content of the molten particle is transferred into the precoat and into the specimen.

The heat from the burning gasses and air turbulence is, however, transferred into the entire length of exposed wire, which promptly increases wire temperature to the vicinity of the gas temperature. This gas temperature has been measured for the power flame spray system to be 1400°F to 1600°F at the spray distance used. For Rokide, this temperature is approximately 600°F. This heating causes the wire to expand and because the wire is held in place at both ends, the wire buckles upward. It also is free to vibrate in the air stream. The gage end loops, which are cantilevered out from the tape may vibrate causing flame sprayed particles to build up beneath the end loops forcing them to curve upward at the ends. Foil gages, or flattened wire gages are more easily bent, because stiffness is reduced. The stiffness is a third order function of the distance from the neutral axis to the outer fiber. Also, flattened wire or foil gages have an increased area against which turbulent air forces can work to initiate vibrations.

The gage wire, held firmly at both ends, buckles upward from heat expansion while the flame sprayed particles fill in beneath the arched wire. A railroad analogy of this phenomenon, on a larger scale, is shown in Figure 22. The figure shows railroad tracks which buckled from heat during a trestle fire. The tracks were held firmly at the cool ends of the trestle. As the wooden trestle burned, the rails became hot and expanded, the thermal expansion forces became large enough to overcome the restraining forces of the rail spikes which gave way and the rails buckled. The same phenomenon happens when flame spraying a strain gage. (Also, the same phenomenon happens when flame spraying lead wires.) When the strain gage grid wire buckles upward from heat, flame sprayed material builds up under the wire. This increases gage thickness, which reduces the strain range and thermal shock resistance of the installation, and changes the gage factor correction for the installation due to increased cement layer thickness. The expanded hot wire is buried in flame sprayed material which eventually cools, but the wire, rigidly encapsulated in ceramic, is restrained from contracting to its original length. The wire cools in a state of residual tension. These residual tensile stresses are very detrimental to gage fatigue life.

The height to which a wire will bow upward from heat is a function of the thermal expansion coefficient of the wire  $\alpha_w$ , the wire temperature increase caused by the hot gasses  $\Delta t_w$ , and the length of the wire between the fixed ends  $\lambda$ . See Figure 23.





FIGURE 22. Railroad Tracks Buckled From Heat During Trestle Fire

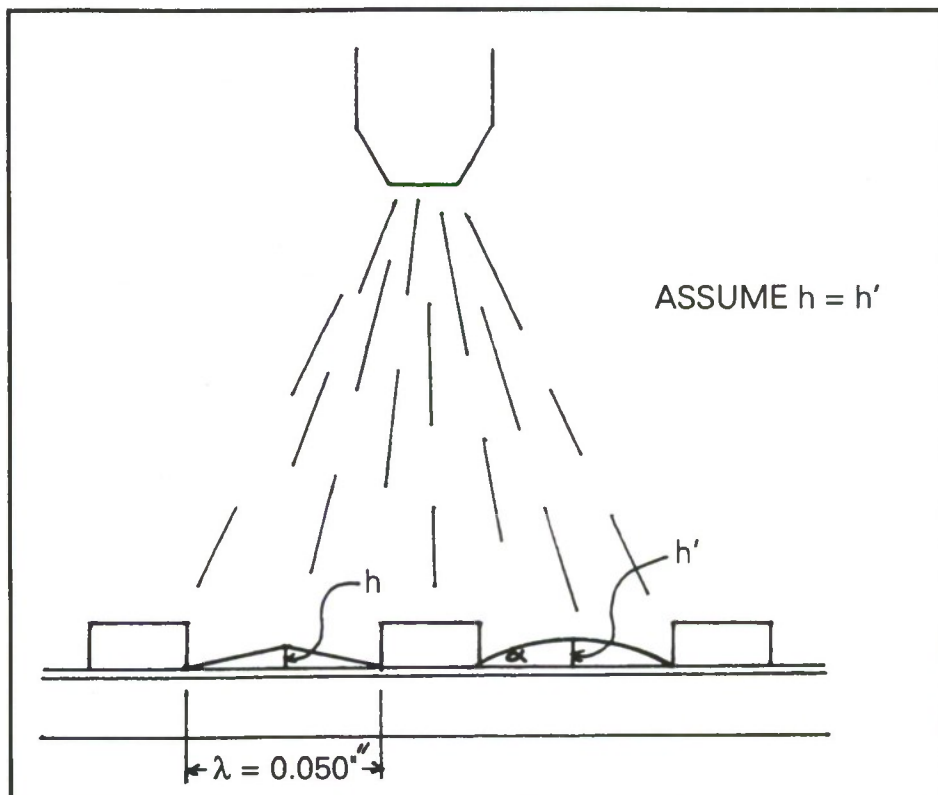


FIGURE 23. Strain Gage Wires Buckle Due to Hot Gases During Flame Spraying

### 3.7.5.1 Sample Calculation

Assume:  $\alpha_w = 4\text{ppm}/^\circ\text{F}$

$$\Delta t_w = 1500^\circ\text{F}$$

$$\delta_w = \alpha_w \times \Delta t_w \times \lambda$$

$$\delta_w = 0.000004 \times 1500 \times 0.050$$

$$\delta_w = 0.00030$$

$$\epsilon_w = \frac{0.0003}{0.05} = 0.006000 \text{ or } 6000\mu\text{m}/\text{m} \therefore \text{above yield stress}$$

$$\cos \alpha = \frac{0.050/2}{0.050/2 + 0.00030/2} = \frac{0.02500}{0.02515} = 0.9940$$

Therefore  $\alpha = 6^\circ$  17 feet; Then  $\sin \alpha = \frac{h}{0.02515}$ ;  $h = 0.0027$  inch

for  $\lambda = 0.050$  inch  
 $\alpha = 4$  ppm/ $^\circ\text{F}$

for  $\lambda = 0.050$  inch  
 $\alpha = 10$  ppm/ $^\circ\text{F}$

$\Delta t_w, ^\circ\text{F}$	300	1,500
h, mils	0.5	2.7
$\epsilon_w \mu\text{m}/\text{m}$	1,200	6,000

$\Delta t_w, ^\circ\text{F}$	300	1,500
h, mils	0.9	4.3
$\epsilon_w \mu\text{m}/\text{m}$	3,000	15,000

for  $\lambda = 0.1$  inch  
 $\alpha = 10$  ppm/ $^\circ\text{F}$   
 $\Delta t_w = 300^\circ\text{F}$   
 $h = 7.7$  mils

for  $\lambda = 0.2$  inch  
 $\alpha = 10$  ppm/ $^\circ\text{F}$   
 $\Delta t_w = 1,500^\circ\text{F}$   
 $h = 34.7$  mils

The above shows there is no substitute for keeping the wires cool! The calculation for  $\lambda = 0.1$  inch and  $0.2$  inch shows the importance of keeping the tape bars no more than  $0.1$  inch apart. Also, if there is any significant mismatch between the thermal expansion of the lead wire and the specimen, the leads must be convoluted or the tape bars will not hold them. As it is, experience has shown that the teflon fiberglass tape will not hold

down any wire larger than 5 mils diameter even if it is convoluted. The teflon aluminum tape will hold down convoluted wires up to 8 mils diameter.

#### 3.7.5.2 Vortex Cooling

To overcome the heating problem, cooling air was experimented with early in Phase I of this program. In Phase II continued work on cooling included evaluation of several vortex type air coolers. These coolers operate on line air and produce cooling air at temperatures about 50°F below inlet air temperature. It was found that setting the spreader nozzle at an angle of 10 to 20 degrees from the gage surface, and operating at 30 psi air pressure would keep the specimen cool. The nozzle was placed as close to the gage as possible. The cooling air flow was directed parallel to the tape bars in order to keep the grid wires bathed in cool air.

The use of cooling air was combined with improved gun handling technique in order to achieve the goal of keeping the grid wires below 400°F at all times. The spray technique is especially important when operating the powder flame spray system because of the hot gas temperature although it also produced improvement in Rokide installations. The new technique is 1) to line up the spray gun at the proper spray distance just off the specimen, and 2) make a rapid pass across the installation. The gun movement must be sufficiently fast to produce minimal heating of the wire. The gun is instantly returned to the ready position where it will dwell for a period of time until the cooling air returns the specimen to its original cool condition. Time is required to cool the specimen from the temperature increase caused by the molten ceramic particles.

#### 3.7.5.3 Temperature Measurement During Spraying

The operator can develop technique through practice. The simplest check on proper spraying procedure is to install a free filament gage or resistance thermometer and measure sensor temperature on a suitable recorder, such as a X-Y plotter. Figure 24 is a record of temperature during initial (tack) and final (cover) coat of a Pt10 RTD resistance thermometer. A 100-seconds/inch time base is used on the X-axis. After the tack coat, the specimen was placed on a hot plate and heated up to 200°F. The specimen temperature was checked with a thermocouple. Upon cooling, the plotter was rezeroed mid-scale and the temperature of the cover coat recorded. Since about 50% of the grid was exposed to the flame, the sensor temperature is estimated to be about two times that shown on the recorder. In this case the sensor temperature was actually about 400°F. This is about right for a good installation, however, the cooler the better. As the sensor heats up it expands. If the sensor is locked in by the ceramic while it is very hot and expanded, it will be locked in residual tension. The higher the residual tensile stresses in the grid, the lower the fatigue life of the installed sensor. Therefore, for good sensor life, keep the passes quick and allow plenty of cooling time. Pt8W gages installed



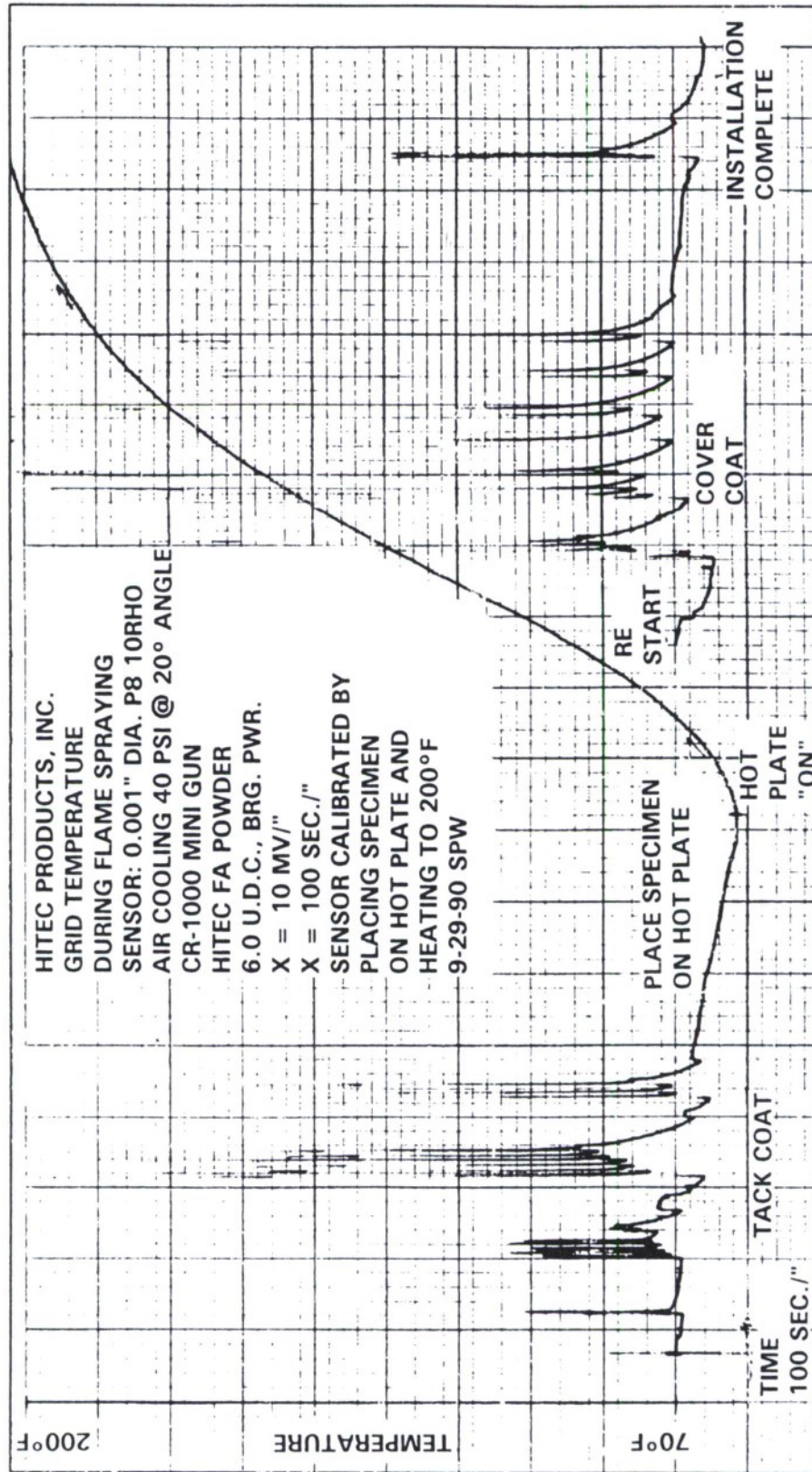


FIGURE 24. Sensor Temperature During Flame Spraying

properly will run a minimum of 1 million cycles at  $\pm 1500\mu$  inch. Another indication of proper cooling is the absence of tape residue after tape removal. Any discoloration or visual residue from the tape carrier is an indication of overheating.

#### 3.7.5.4 Coating Thickness

Heat dissipation becomes more difficult as a coating gets thicker and thermal stresses within the coating build up. The proper thickness should be as follows:

A. Precoat	1-2 mils
B. Tack Coat	2-4 mils
C. Cover Coat	2-4 mils
Total:	6-10 mils (Typical)
Never to exceed:	16 mils

As a visual indication, the precoat should be stopped before it appears all white. The tack coat should be stopped before all the lead is covered. The lead should only be partially covered, just enough to keep the lead in place during tape removal. Any sharp edges should be broken at a 45° angle at the tape bar edge. Use a very sharp pick and press lightly on edge of coating near tape bar edge. Do not touch grid wire. Blow off all ceramic and tape residue. Remove all tape adhesive residue with sharp tweezers. The use of the quad tape method shown in Figure 21 and described in section 3.7.4.4.3 should eliminate most of the need to break sharp corners. The cover coat should be just sufficient to cover grid and leads. An outline of the grid and leads should be clearly evident. Blend all sharp corners from masking tape using a white aluminum oxide stone.

#### 3.7.5.5 Uncoated Carbon Composites

##### 3.7.5.5.1 Strain Gage Ceramic Cements

Bonding to uncoated carbon composites is relatively easy using ordinary strain gage ceramic cements and standard procedure. The installations are generally usable to 1200/1500°F for short term testing. Strain gages have been run up to 1700°F for brief periods and have survived, but electrical leakage within the cements generally limit them to the lower temperatures of 1200/1500°F.

##### 3.7.5.5.2 Modes of Failure Dictate Gage Design

There are two basic modes of failure: A) oxidation and volatilization of the layer of carbon to which the cement is bonded, and B) shear fracture of the ceramic cement or ceramic/carbon interface due to thermal expansion stresses between the cement and the gage. Because carbon composites have a near zero thermal expansion, and strain gage ceramic cements have been designed to work on metals with a 6-9 ppm/°F expansion,

the thermal mismatch causes large shear stresses at the bond line. These stresses can be minimized by keeping the cement layer as thin as possible. Increasing cement thickness increases the bond line shear stresses. Thick cement installations will fracture at the bond line and spall off. Keeping cement as thin as possible has always been good practice and is essential on carbon composites.

Additional stresses are introduced in the cement by the imbedded strain gage and lead wires. The metallic strain gage expands thermally more than the carbon and must be restrained by the cement. The amount of thermal stress introduced by the strain gage depends on:

- A) wire cross-sectional area
- B) wire modulus of elasticity
- C) strand spacing
- D) thermal expansion coefficient
- E) temperature change,
- F) all of the above for the lead wire

Stresses from the gage and leads are reduced as a square of the wire diameter. Stresses will also be reduced proportional to reduction in modulus of elasticity. However, the modulus is nearly the same for most high temperature strain gage materials. The strand spacing is important. Gages with a fine pitch (0.005 inch spacing) cause twice the shear stress of gages made with a wide pitch (0.010 inch strand spacing).

The thermal expansion coefficient of the gage wire should be as low as possible. Noble metal alloys are much better than nickel or iron based alloys with their much greater thermal expansion coefficients.

In summary, in order to minimize stresses within the cement caused by the gage and leads, select gages with:

- 1) a small wire diameter\*
- 2) increased strand spacing (wider pitch gages)
- 3) Use less strands. Use gages having lower resistance values and longer gage length. (This also is preferable for composites made from woven fabrics.)
- 4) Use gage alloys having a lower thermal expansion coefficient.
- 5) Use the smallest diameter lead wire possible and convolute the leads.

\*There is a trade off with gage life and stability. As the wire diameter is reduced, the ratio of surface area to material volume increases, and oxidation effects cause resistance drift to increase.



#### 3.7.5.5.3 Convoluting Leads

Lead wires represent a major inclusion which generates stresses in the cement to a much greater extent than the gage grid. One method for reducing these stresses is to reduce lead wire diameter. Another method is to put the leads in a bending mode rather than a pure columnar tensile or compressive mode by convoluting the leads. A gage design employing the principals discussed above is shown in Figures 25, 26, 27, and 28.

#### 3.7.5.5.4 Low Expansion Cements

Through 40 years of ceramic cement development most strain gage work was done on metals, and the cements were designed to be compatible with metals. This called for cements with high thermal expansion and other features to help them bond well to metals. The principal bonding agents in strain gage ceramic cements are phosphoric acid or aluminum dihydrogen phosphate. Either of these acids, when used directly on metals, react with the metal generating copious amounts of hydrogen gas, which tends to bubble in the cement and produce porous coatings full of voids. Additions of chromic anhydride to the phosphoric acid binder greatly reduce gas generation and may enhance the bond to metals as well. For bonding to carbon composites (or for many ceramic materials as well) reaction with the specimen is not a problem, and therefore the addition of chromic anhydride is not required. (Chromic anhydride is a known carcinogen and its elimination from cements would be welcomed by the formulator as well as by the user.)

Experimental cements formulated for sensor applications<sup>2,3</sup> on carbon composites and ceramics consisted of alumina based White Cerro (WC-1 through WC-12) and used aluminum dihydrogen phosphate as the binder. These cements should be restricted to work with noble metal sensors as hydrogen gas generation can still be a problem with some reactive sensor alloys.

WC-9 has been evaluated on this program for bonding temperature sensors on carbon composites. No excessive gas generation was noted in bonding 0.005 inch dia. bare type K wires to carbon composites. The type K TC's were found to stay bonded with two 5 minute excursions to 2100°F. The WC-9 also protects the carbon layer to which it is bonded from oxidation, while unprotected carbon is severely attacked.

With the carcinogen eliminated, application of WC-9 by air brush was experimented with. Nice thin coatings were achieved, but adherence to the carbon was not nearly as good as brush applied coatings.

#### 3.7.5.6 Flame Sprayed Coatings

Initial work with powder flame sprayed coatings revealed that carefully applied coatings to carbon composites could withstand short term testing to 2000°F. This fact combined

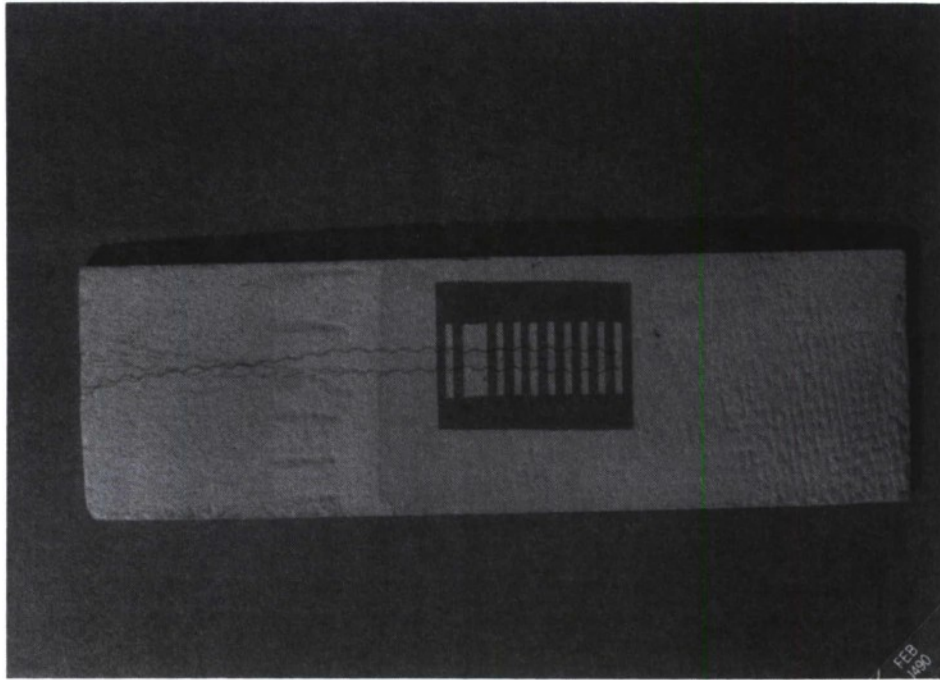


FIGURE 25. A PT8W strain gage with convoluted leads is positioned on a carbon composite. The composite is coated with 3 to 4 mils of flame sprayed FA powder.

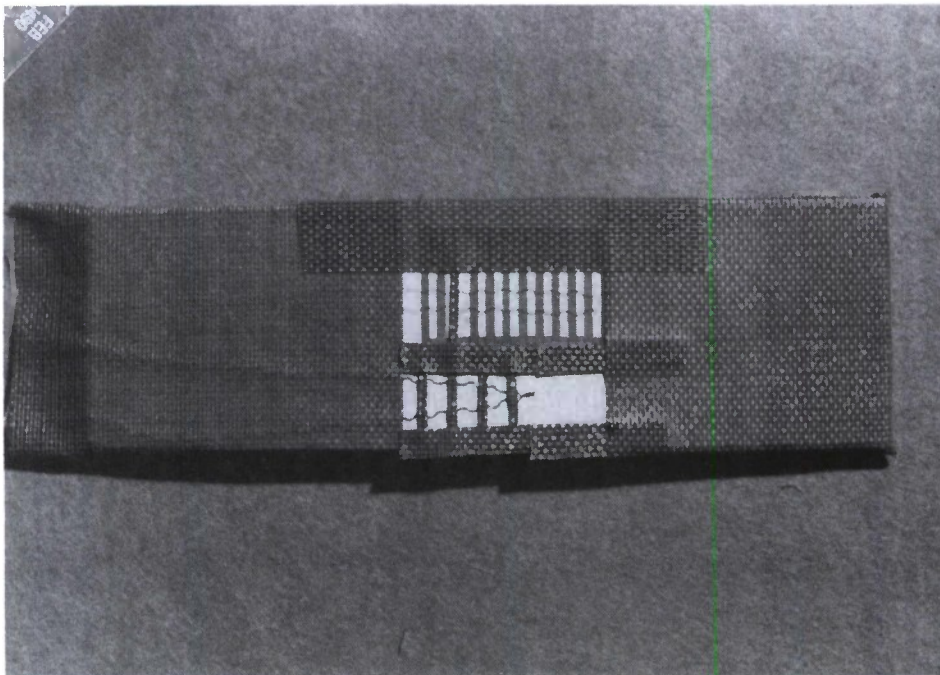


FIGURE 26. A 5-mil-dia. convoluted type S thermocouple is taped in place next to the strain gage. The gage and thermocouple are boxed in using No. 64 tape to prevent excess overspray on the specimen.



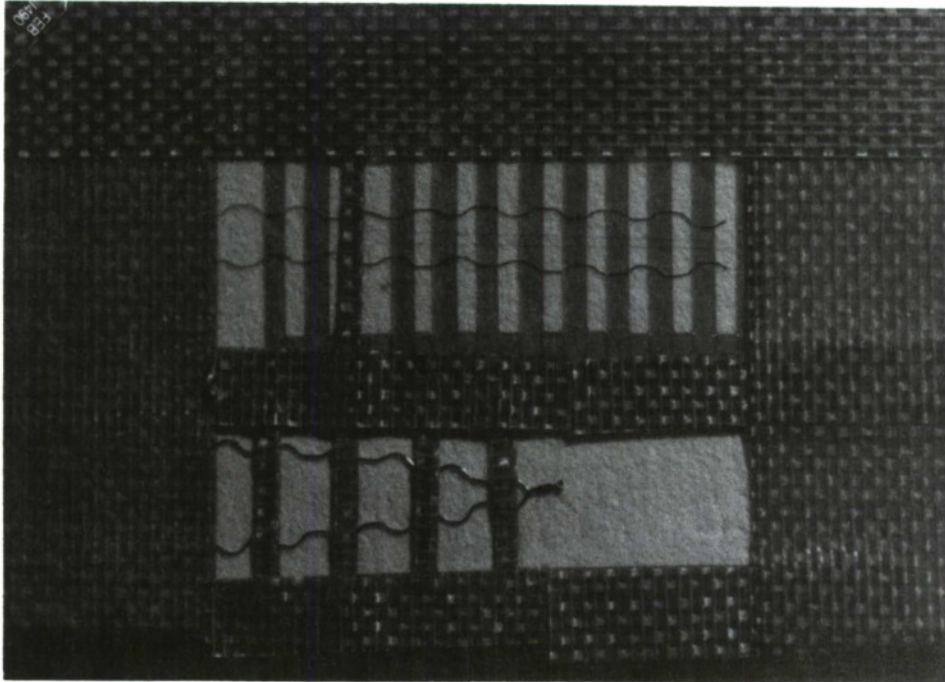


FIGURE 27. Close-up of Gage and Thermocouple

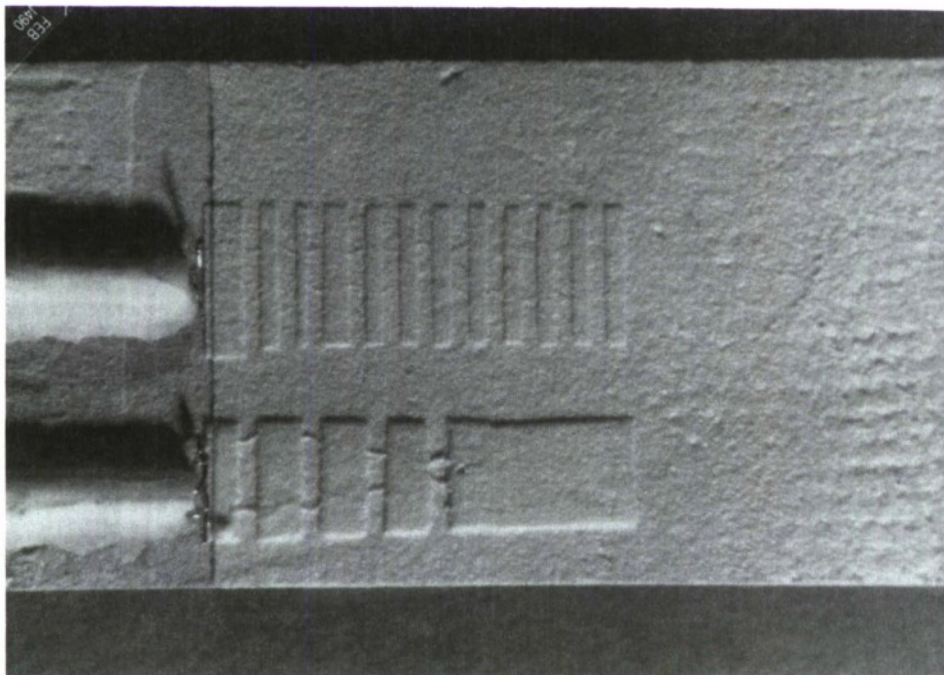


FIGURE 28. Close-up of Completed Gage and Thermocouple Installation on Carbon Composite Specimen. Note, extension leads are routed through two hole alumina insulators, which are bonded to the specimen with WC-9 cement.



with the excellent electrical properties of Hitec FA high purity alumina powder<sup>3</sup> made this attachment system an attractive candidate for further development.

The pull tests indicated bond strengths to strain gage leads and to substrates equal to the best ceramic cements, however, inferior to the bond strengths of Rokide. With a gage design for composites having convoluted leads of 0.002 inch diameter platinum 10 nickel it was found during pull tests that all convoluted leads would break rather than pull out of a powder sprayed test sample.

Certain design improvements in the CR-1000 Mini-Gun flame spray system, namely 1) a redesign in the combustion hardware to improve ceramic sprayability, and 2) a redesign minihopper to allow small quantities of experimental ceramic mixtures to be changed quickly made it readily adaptable for development of new powder mixtures.

One of the major differences in using the powder flame spray compared with Rokide is in the specimen and sensor temperature during application. Rokide is a relatively cool procedure, with the sensor and specimen remaining relatively cool during normal application, i.e. less than 130°F. With the powder spray, however, the sensor and specimen are located at the tip of the flame, and both sensor and specimen can become extremely hot quickly. To achieve sensor bonding without burning carrier tapes, very quick passes were required with long pauses between passes to allow the part to cool. This procedure exceeds the patience of most technicians, especially when working on programs with deadlines.

Air cooling was experimented with on this program and found to speed up application time. Thirty psi air was introduced at a 10- to 20-degree angle to the specimen and directed across the gage grid worked very well. Vortex air coolers were tested and found to work better than line air. Vortex air coolers provide air at about 50°F below line air temperature. This device further improved application speed to nearly equal that of the Rokide process. A desiccant filled (silica gel) air filter in line with the cooler was necessary to eliminate all oil and moisture contaminants in the air supply. Contaminated cooling air is one cause of delamination in powder spray coatings. Another cause of delamination is excessive heating of the coating by slow passes or excessive dwell of the flame on the coating.

#### 3.7.5.7 Tests on Carbon Composites

Not all carbon composites can be gaged. On those composites which are impregnated with various materials, it is extremely difficult to adhere ceramic cements or flame sprayed coatings. See Figures 29 and 30. Pure carbon composites used on rocket nozzles are relatively easy to bond ceramic cements or powder flame sprayed coatings. The pure specimens were used in the following tests.

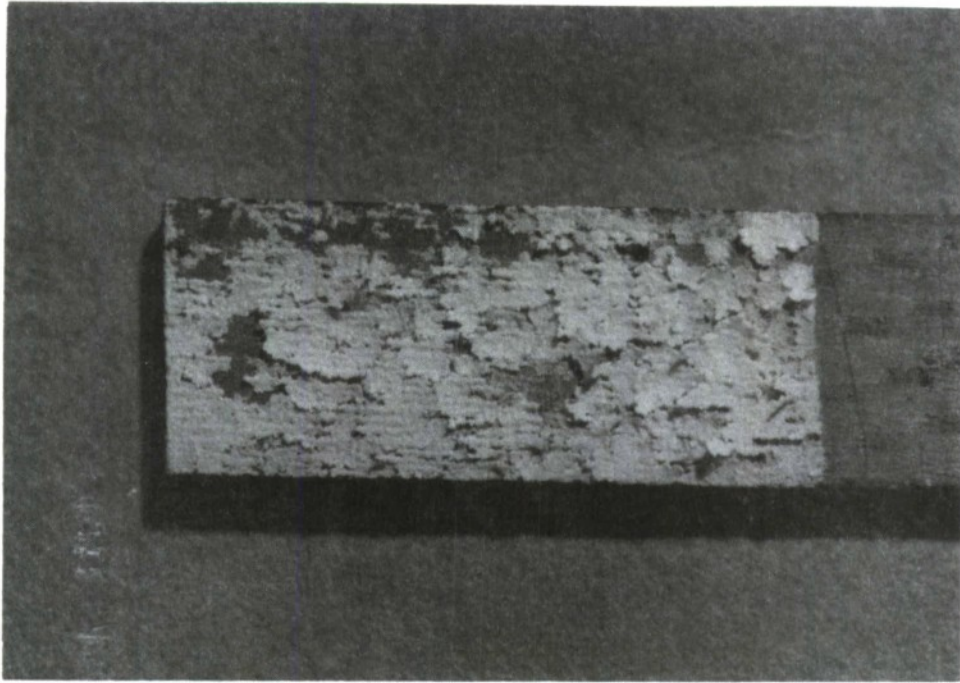


FIGURE 29. Not all carbon composites can be coated with ceramics. Powder flame sprayed alumina would not adhere to this specimen.

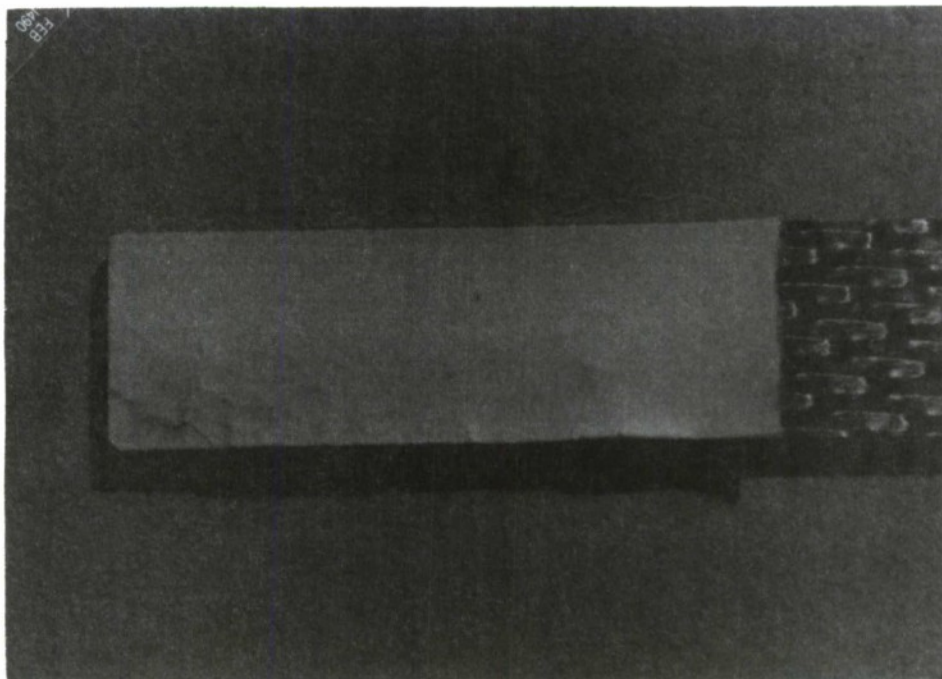


FIGURE 30. Powder bonding of a powder flame sprayed alumina coating is shown on this specimen.



Two tests were conducted to 2550°F on 0.080 inch x 1/2 inch x 4 inches Thiokol<sup>2</sup> specimens. The first specimen was coated all over with 3-4 mils of FA powder. A Pt8W gage with 2 mil convoluted and flattened Pt Ni leads was installed using FA powder and CR-1000 Mini-Gun. A 5-mil convoluted type S thermocouple was similarly installed. (See Figures 25, 26, 27, and 28.) Two hole AD-998 alumina insulators were bonded to the specimen using WC-9 cement and cured to 650°F. Platinum leads were spot welded to the gage and fed through the insulators (see Figure 31). The gage output was recorded on the Y-axis of an X-Y plotter and the temperature was recorded on the X-axis. The specimen was plunged into a preheated furnace and the gages opened about half way up in temperature. The thermocouple remained intact to 2606°F maximum temperature. Room temperature examination revealed numerous cracks in the coatings. (See Figure 32)

The second specimen was not coated all over with alumina powder. The gage (new design) was similarly installed using FA powder and the CR-1000 Mini-Gun. The alumina feedthroughs were attached to the specimen using WC-9 cement for about 1/2 inch length and Aremco 551A carbon cement for the remaining length. See Figure 33. This specimen was similarly tested by plunging into a preheated furnace. The resistance temperature curve is shown in Figure 58. At almost maximum temperature the lead tube debonded and the specimen fell into the furnace. The furnace was shut off. About two hours later the specimen was removed and examined. The gage and thermocouple were operational. The gage resistance measured 108.6 ohms at room temperature. Therefore, the gage and thermocouple installations work to 2600°F, but the lead wire insulator attachment needs work. The 551A carbon cement works well to about 2100°F, but becomes very weak at higher temperatures.

Figure 34 shows a 5 mil type K thermocouple bonded to carbon composite with WC-9 cement. The thermocouple melted at 2550°F. Some of the spheres of chromel and alumel are still evident in the photograph. The WC-9 cement had sufficient strength to stay on the specimen.

#### 3.7.5.8 Silicon Carbide Coated Carbon Composites

The procedure for bonding to SiC coated C/C found usable for short excursions to 2100°F was to A) coat the silicon carbide with a 4 to 6 mil precoat of SC cement, B) cure 24 hours at room temperature, C) cure for 1 hour at 600°F, D) install gage or thermocouple using Rokide or powder flame spray directly to the SC cement coating. Thermocouple or gage wire leads should not exceed 5 mils in diameter and should be convoluted.

A new design platinum 8 tungsten gage and a 5-mil Pt/Pt10Rho thermocouple were installed on a 1 inch square specimen over an SC cement precoat. The sensors were installed using Hitec FA powder in the CR-1000 Mini-Gun Flame Spray system. (Figures 35 through 39 are step-by-step installation photographs.) The 1/16 inch diameter two hole alumina insulators were bonded to the specimen with SC cement. See Figure 40.



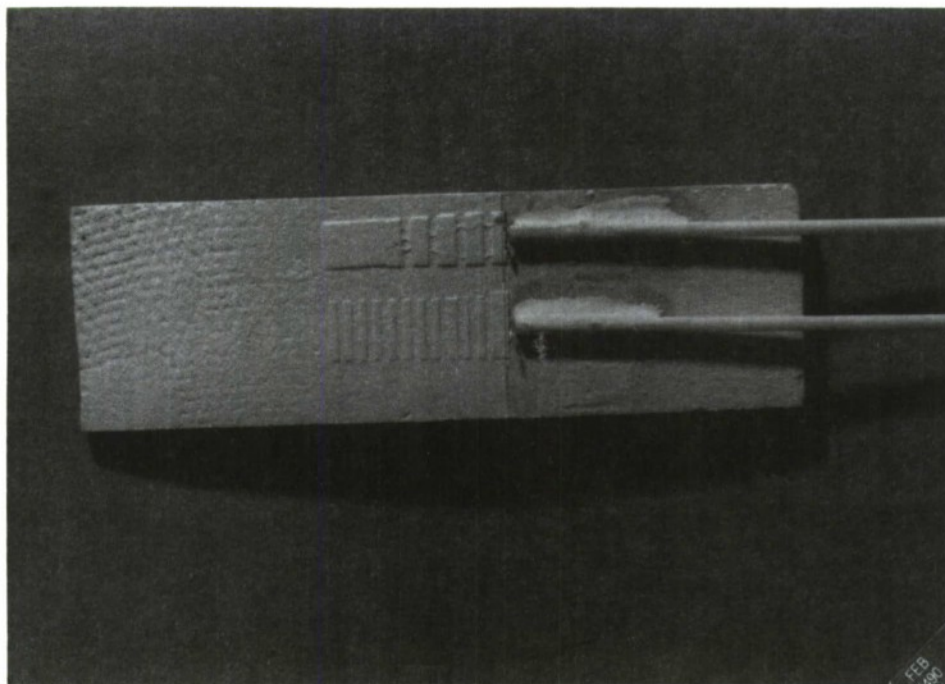


FIGURE 31. Overview of Completed Installation on Carbon Composite Showing Two Hole Alumina Insulators Bonded to the Specimen with WC-9 Cement before Test.

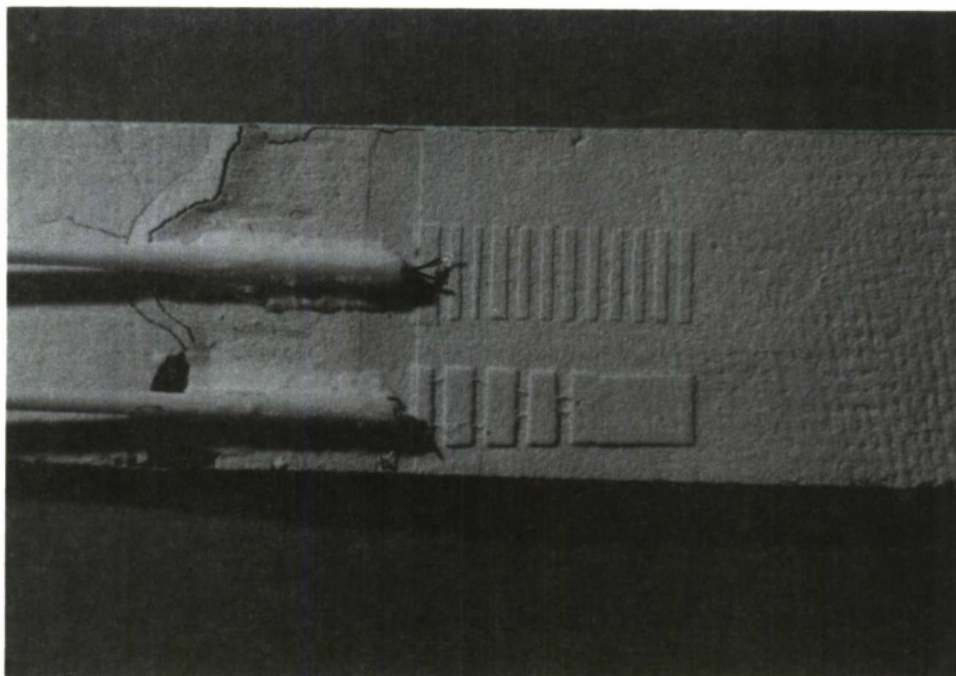


FIGURE 32. Carbon Composite Test Specimen After Plunge Test to 2606°F

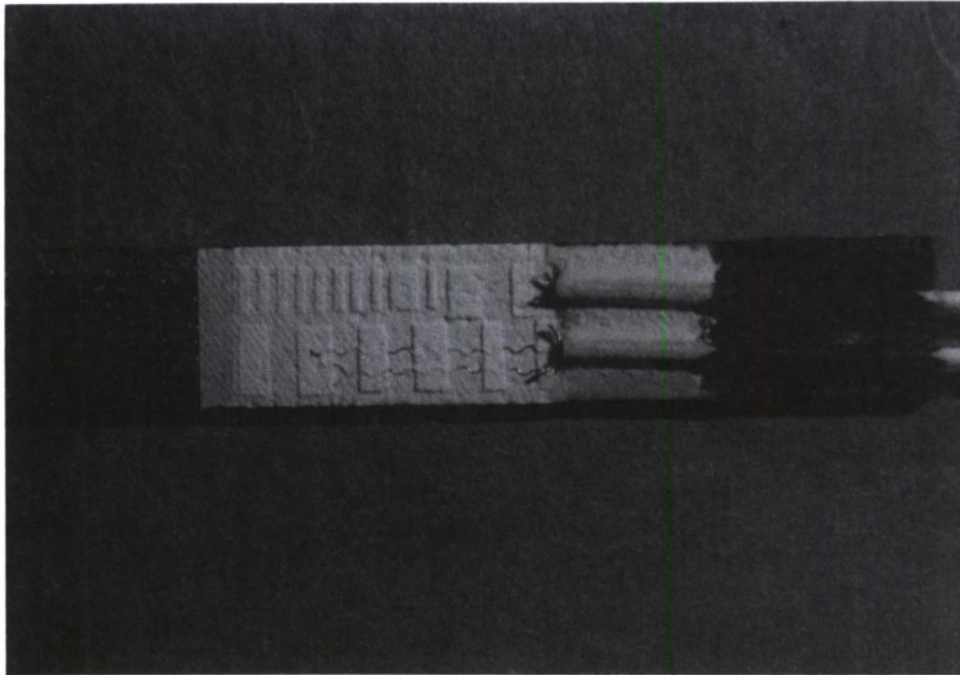


FIGURE 33. Pt8W Gage and 5 mil Convolute Type S Thermocouple Bonded Directly to C/C using FA Powder Flame Spray. Alumina insulators are bonded with WC-9 cement and Aremco 551A carbon cement.

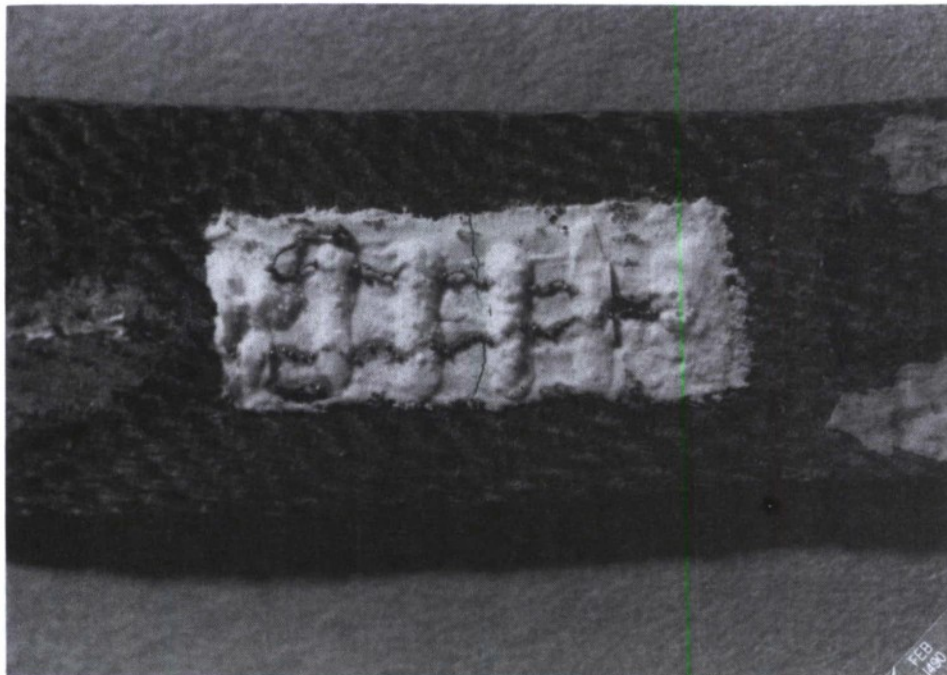


FIGURE 34. A 5-mil-diameter convolute type K thermocouple bonded with WC-9 cement withstands furnace plunge to 2500°F. Note the remains of the thermocouple are a series of small spheres of melted chromel and alumel material.

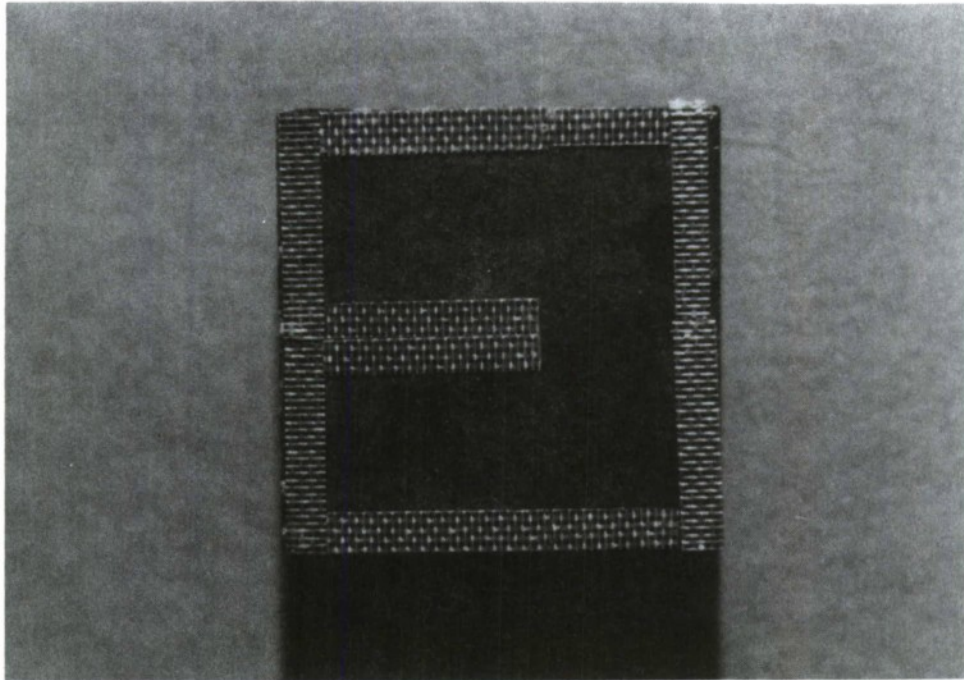


FIGURE 35. Silicon carbide coated carbon composite is masked using 3M No. 64 tape. The tape is used to control cement precoat thickness as well as defining the precoat area.

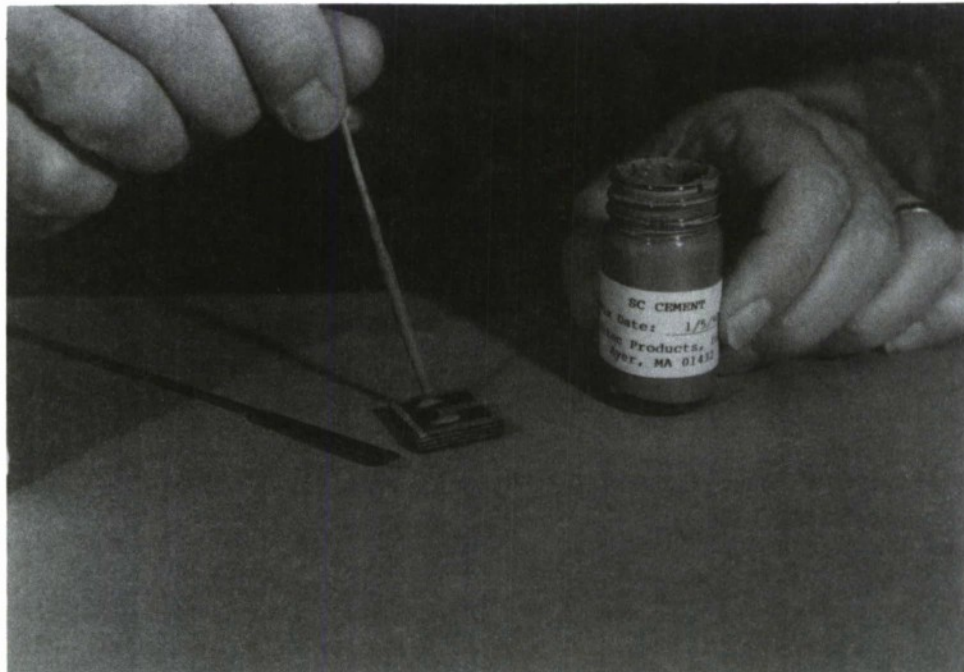


FIGURE 36. SC cement precoat is applied to silicon carbide coated test specimen.



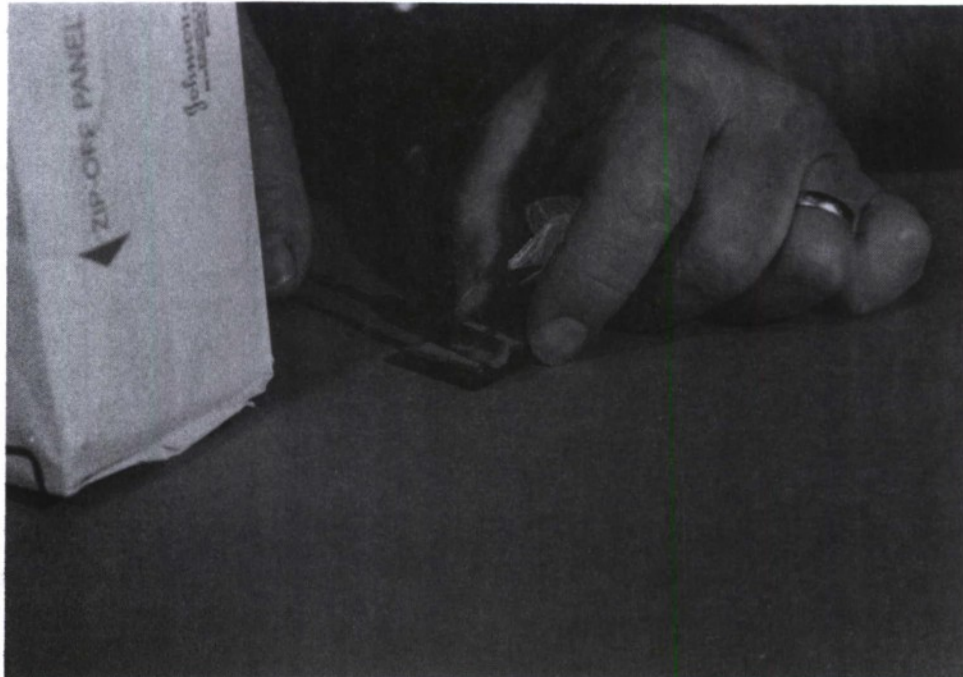


FIGURE 37. A spatula is used to uniformly spread the SC cement over the silicon carbide coated surface.

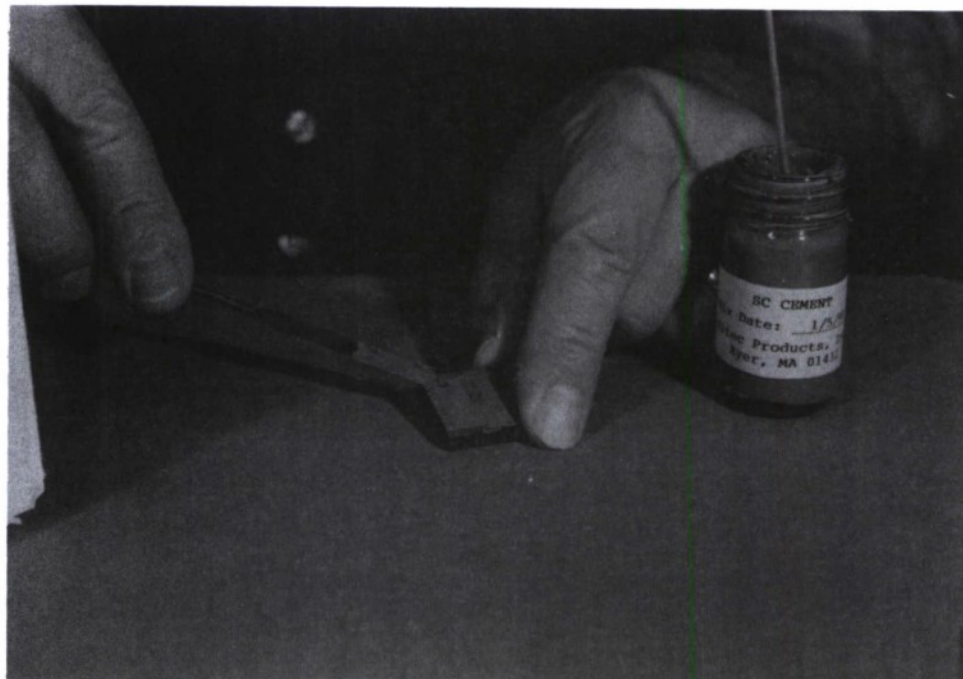


FIGURE 38. Excess SC cement is scraped off even with the tape surface using a stainless steel spatula. Fast work is required as the cement sets in minutes.



FIGURE 39. The strain gage and thermocouple are installed using the CR-1000 Mini-Gun and FA alumina flame spray powder.

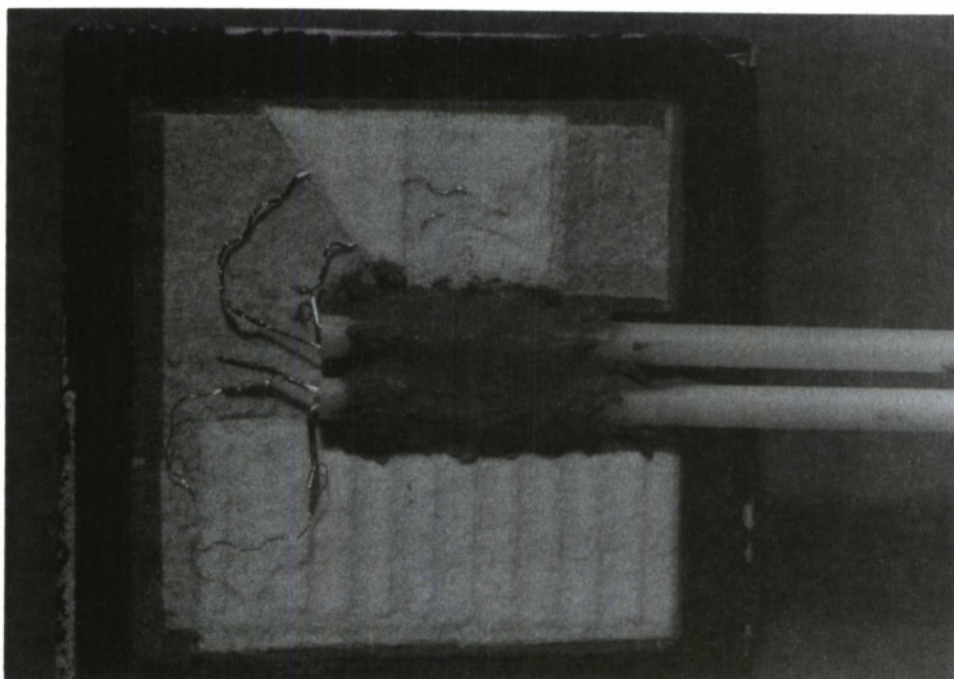


FIGURE 40. Completed Gage and Thermocouple Installation on Silicon Carbide Coated C/C. Note the alumina insulators are attached to the specimen using SC cement.

Ten mil platinum alloy leads were fed through the alumina insulators and spot welded to the gage and thermocouple leads. The assembly was dropped once breaking off the alumina insulators and was quickly repaired with more SC cement. Spatula application of the SC cement inadvertently caused some of the SC cement, which is conductive, to short the sensors (it was discovered during a furnace plunge to 2550°F that the sensors were shorted). No data was taken, but the installation survived totally two plunges to 2550°F.

### 3.7.6 Rene' 41 Tests

The Rene' 41 is a relatively straightforward material on which to apply high temperature instrumentation. It was decided to apply various state-of-the-art strain measuring systems and test them within their useful range. The new PdCr gage being developed by NASA and United Technologies is one such system. The attachment coating consisted of a 96% alumina and 4% zirconia powder applied with the CR-1000 Mini-Gun. A Hitec HDA dual element strain gage, developed under Air Force contract<sup>24</sup> in 1974, which operates in the temperature range of 900°F -1100°F, was attached using FA powder applied by the Mini-Gun. A prototype GPD capacitive strain gage<sup>25</sup> was attached to a Rene' 41 test bar by spot welding and tested up to 2000°F. The new design Pt8W, a Chinese 800°C gage<sup>26</sup> and a FeCrAl foil gage (ME-2 type NZ 2104)<sup>3</sup> were installed using FA powder and the CR-1000 Mini-Gun and tested to the melting point of the Rene' 41.

#### 3.7.6.1 GPD Low Profile Capacitive Strain Gages

Two prototype model 3 GPD capacitive strain gages<sup>25</sup> were fabricated for this program. Hastelloy X was used for the body, plates and ground plane, and flame sprayed Rokide Hitec HT as insulation. This new design has a 1/2 inch square body and a 3/4 inch gage length between attachment points. Overall height was 1/16 inch. (See photo, Figure 41.) The gage was installed by spot welding to a Rene' 41 test bar. Standard Boeing type terminals and cables were used. A 20-gage Nextel insulated type K thermocouple was spot welded to the bar next to the gage. Standard instrumentation consisting of a Hitec Products model 2004 signal conditioner and model 2003 power supply were used for the tests. (See Figure 42.) Gage output was recorded on the Y-axis of an X-Y plotter. The thermocouple output recorded test bar temperature on the X-axis.

The second cycle apparent strain which is identical to first cycle is shown in Figure 43. Total capacitance is recorded during heat up to 2000°F. The significant feature of this test was the absence of amplifier "saturation" which indicates electrical breakdown of the gage. A third run to 2000°F was made with exactly the same results as recorded for cycle two.



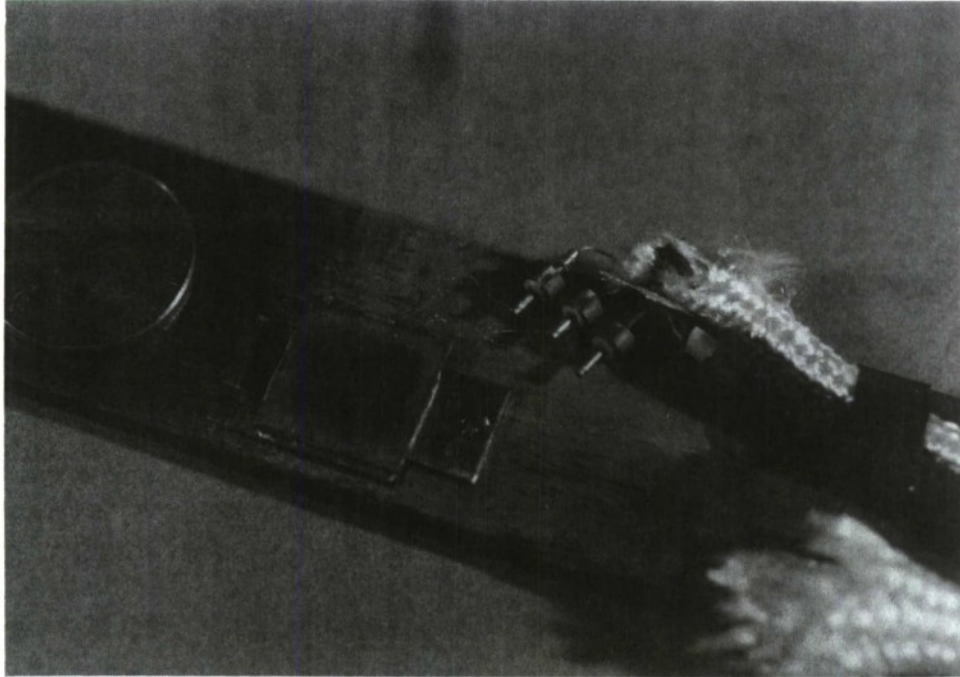


FIGURE 41. Low Profile GPD Capacitive Strain Gage Spot Welded to Rene' 41 Test Bar

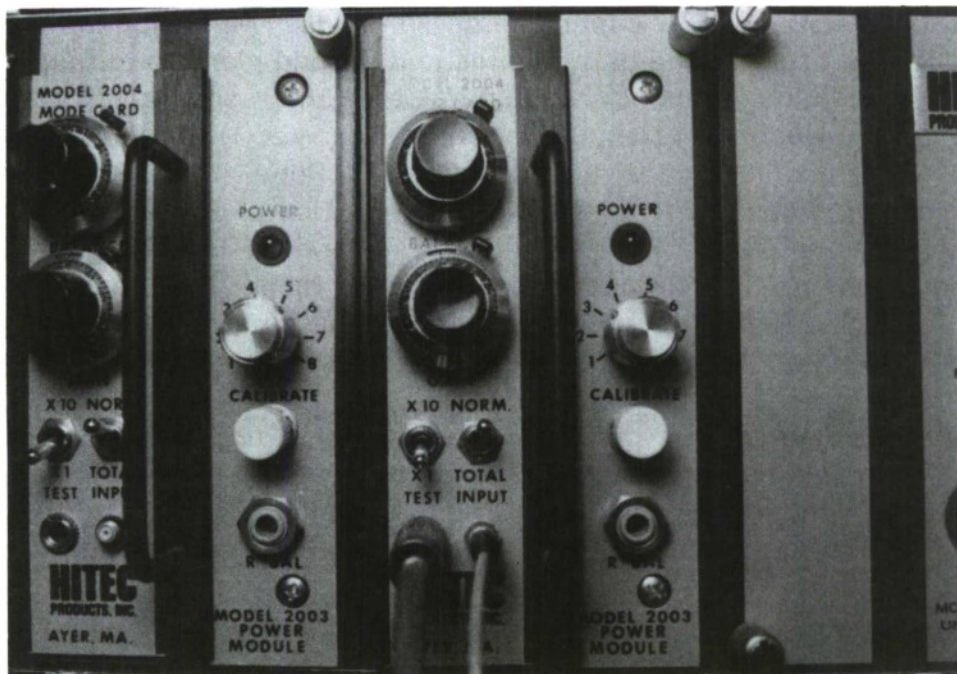


FIGURE 42. Capacitive Instrumentation

Hitec Products Inc.  
 Model 3 GPD Capacitive Strain Gage  
 SN 001 on Rene' 41 Bar  
 Run #2  
 Plunge into 2100°F Furnace  
 Model 2003 Power Supply  
 Model 2004 Mode Card  
 12-13-89 SPW,VPW

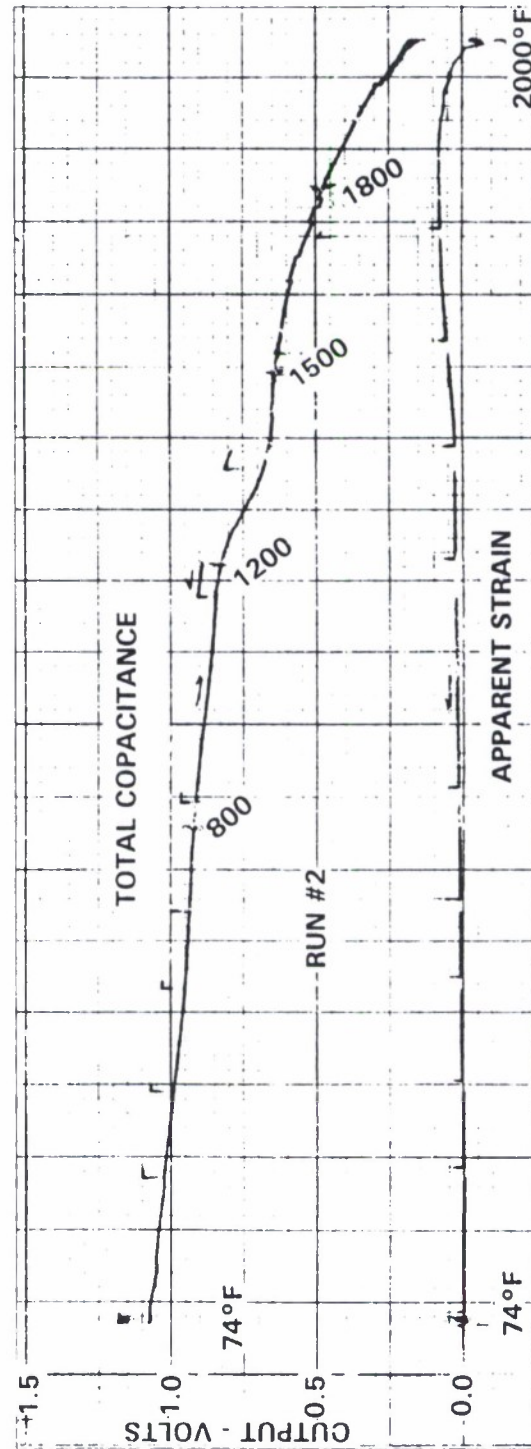


FIGURE 43. Apparent Strain and Total Capacitance vs Temperature for GPD Capacitive Strain Gage

### 3.7.6.2 High Temperature Rene' 41 Tests

Three gage types were tested up to the melting point of the Rene' 41. These were:

- 1) the new design Pt8W Gage,
- 2) the Chinese 800°C Alloy Gage, and
- 3) the FeCrAl Foil Gage (ME-2 Type NZ 2104).

The surface preparation consisted of a no. 60 aluminum oxide grit blast followed by a standard FA powder flame spray application using the CR-1000 Mini-Gun. The vortex cooler was used to provide cooling air. A type S thermocouple was installed next to the gage. Ten-mil platinum leads were spot welded to the gage and fed through two hole AD-998 alumina insulators 6 inches long. The alumina insulators were attached to the specimen using Hastelloy X straps spot welded to the Rene' 41 specimen. (See Figures 44 through 46.)

The type S thermocouple was read out on the X-axis of an X-Y plotter and simultaneously on an Omega Engineering digital indicator. The strain gage was powered by a 6.0 volt d.c. powder supply. General Radio decade resistors were used to complete the bridge. Calibration was achieved by adjusting the decade resistor over the range of interest.

The first gage to be tested was the Pt8W gage compensated by a platinum thermometer. It was placed into a cold furnace and heated up slowly. The furnace employed silicon carbide heating elements and an Omega Engineering controller (See Figures 48 and 49.) A 3/8 inch x 2 inches cut-out in the top of the furnace was used as an access port. The cool end of the alumina insulators outside the furnace were clamped between two 1 inch x 4 inches x 3/8 inch alumina felt pads which served to seal the furnace opening when the specimen was fully inserted into the furnace. A Hargrave clamp held the felt firmly in place and served as a handle to pull the specimen from the furnace when required.

Figure 50 is a record of gage resistance vs. temperature up to 2500°F. Note the thermometer element failed at about 2300°F.  $R_b$  was readjusted at about 2430°F. Upon reaching 2500°F the specimen was promptly withdrawn by lifting on the leads. It was discovered that the specimen had melted and swelled at its base and would not come out through the furnace opening. It was finally forced through the opening, but the alumina feedthroughs partially lifted from the specimen breaking the gage leads. See Figure 47.

The second Rene' 41 specimen was inserted into the preheated furnace. This was the Chinese 800°C gage and it was withdrawn after reaching 2451°F and cooled to room temperature. The specimen was also melted about the same amount as the first specimen. The plotter was re-zeroed and the Y scale amplified by ten times for the



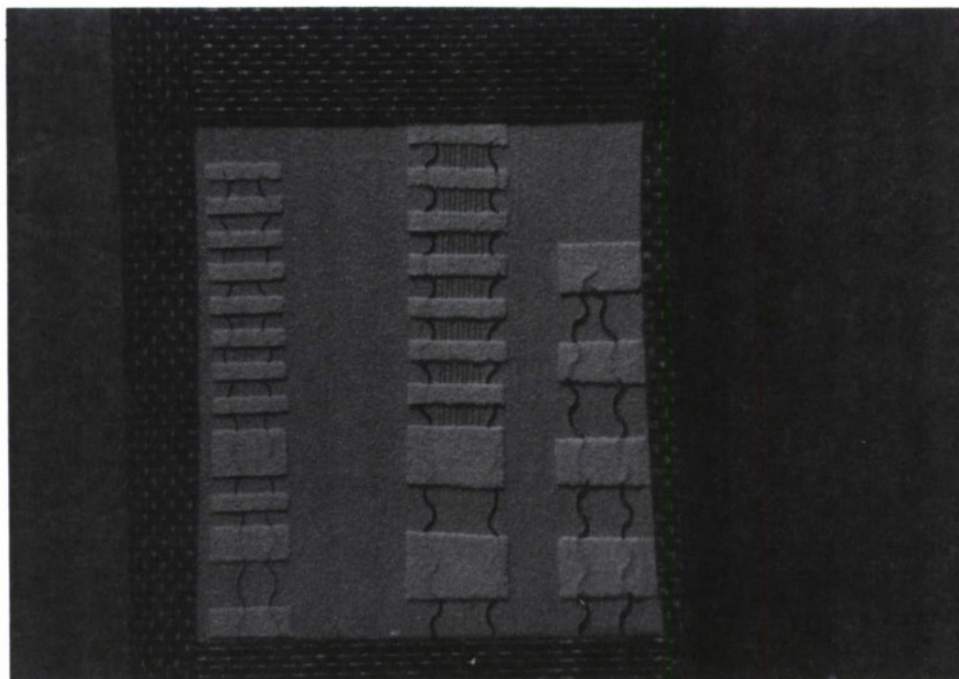


FIGURE 44. Pt8W Gage (left), Pt Resistance Thermometer (center), and Type S Thermocouple (right) installed with FA Powder Using the CR-1000 Mini-Gun. Initial Tack coat is shown.

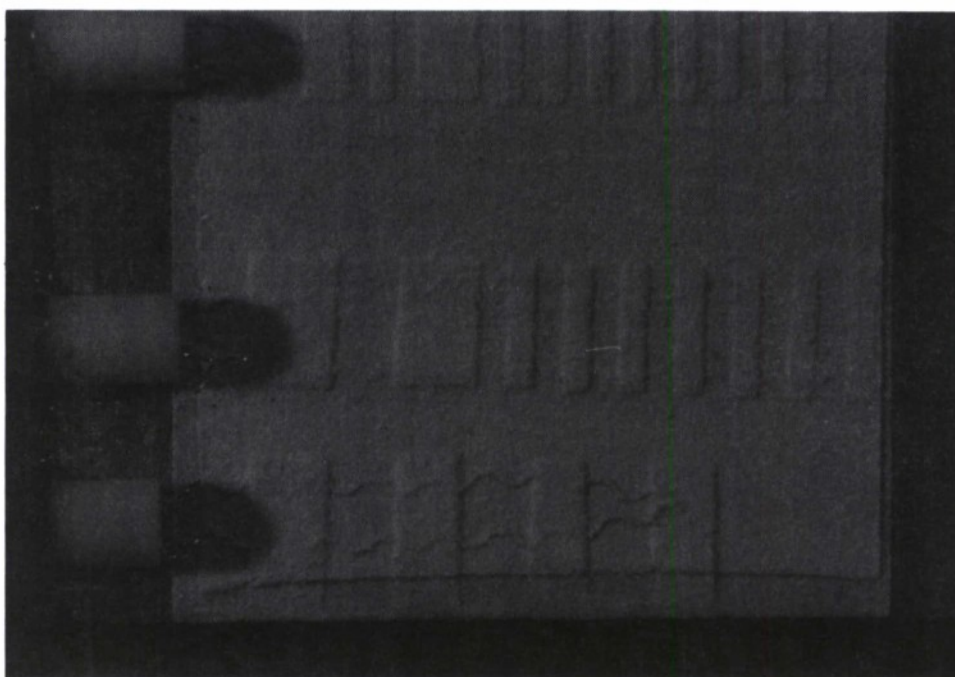


FIGURE 45. Close-up of Completed Pt8W Installation on Rene' 41

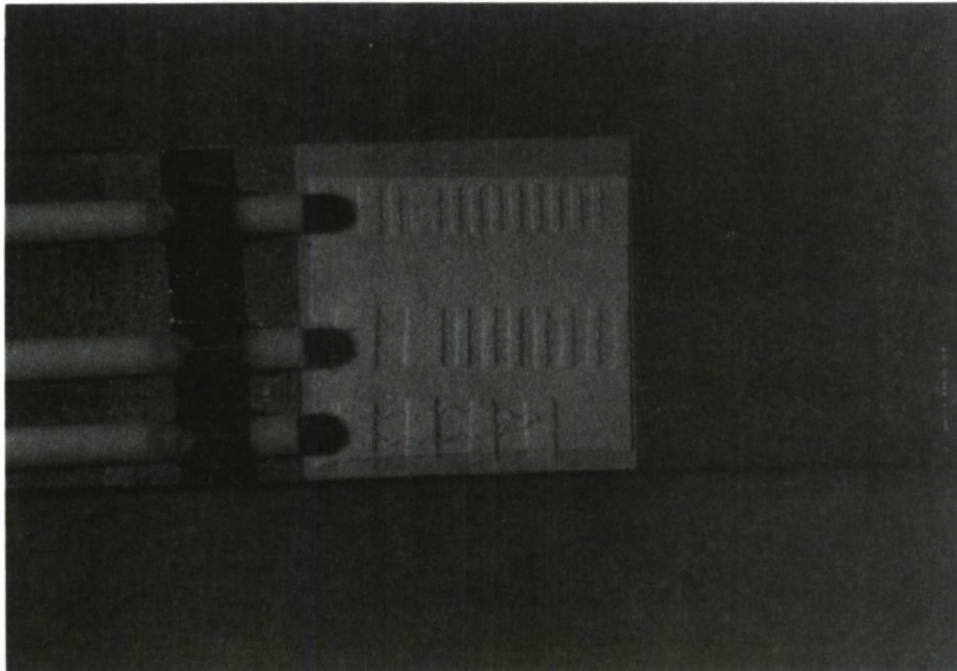


FIGURE 46. Overview of Completed Pt8W Installation on Rene' 41

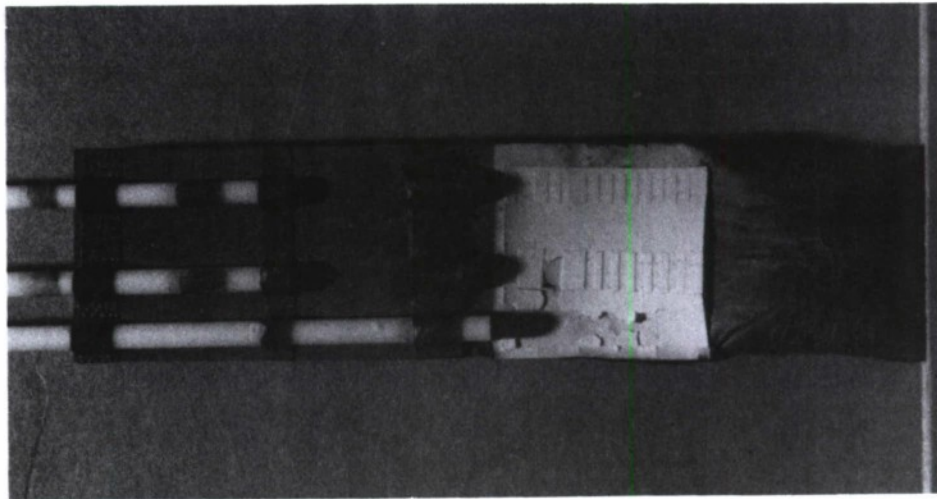


FIGURE 47A. Figure 46 Pt8W Installation After Test. Note specimen melted introducing considerable flow strain into the installation.

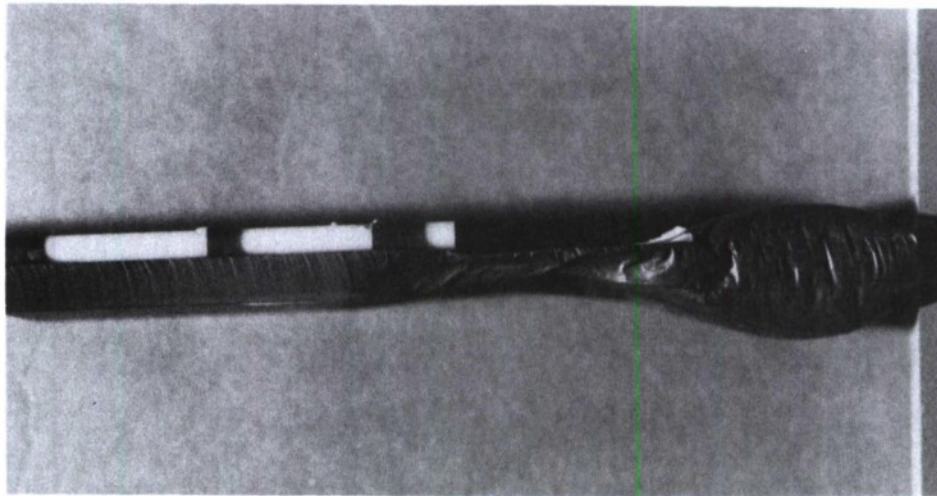


FIGURE 47B. Side View of Melted Specimen

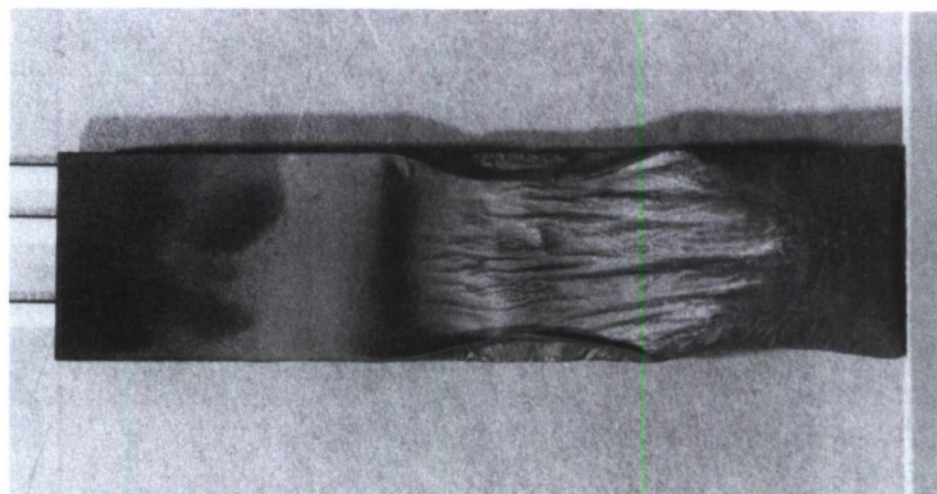


FIGURE 47C. Back View of Melted Specimen



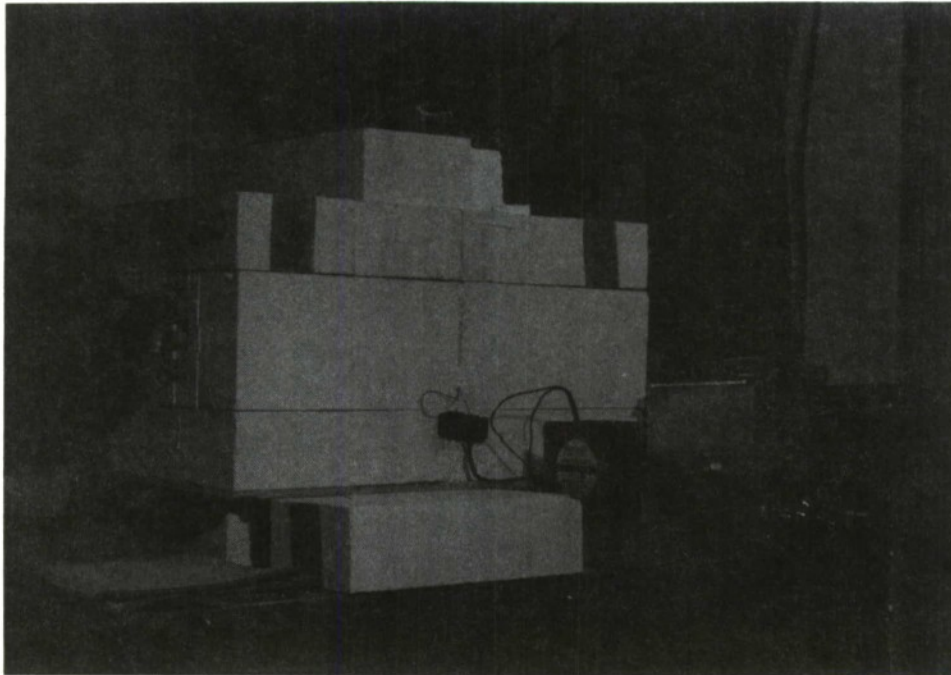


FIGURE 48. High Temperature Furnace and Controller

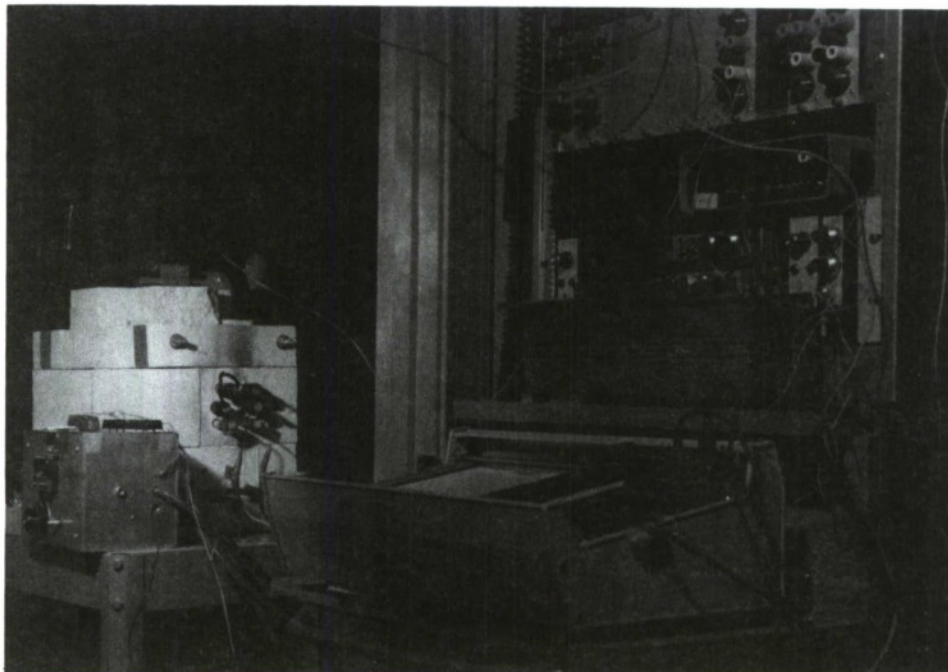


FIGURE 49. High Temperature Furnace, Controller and Instrumentation

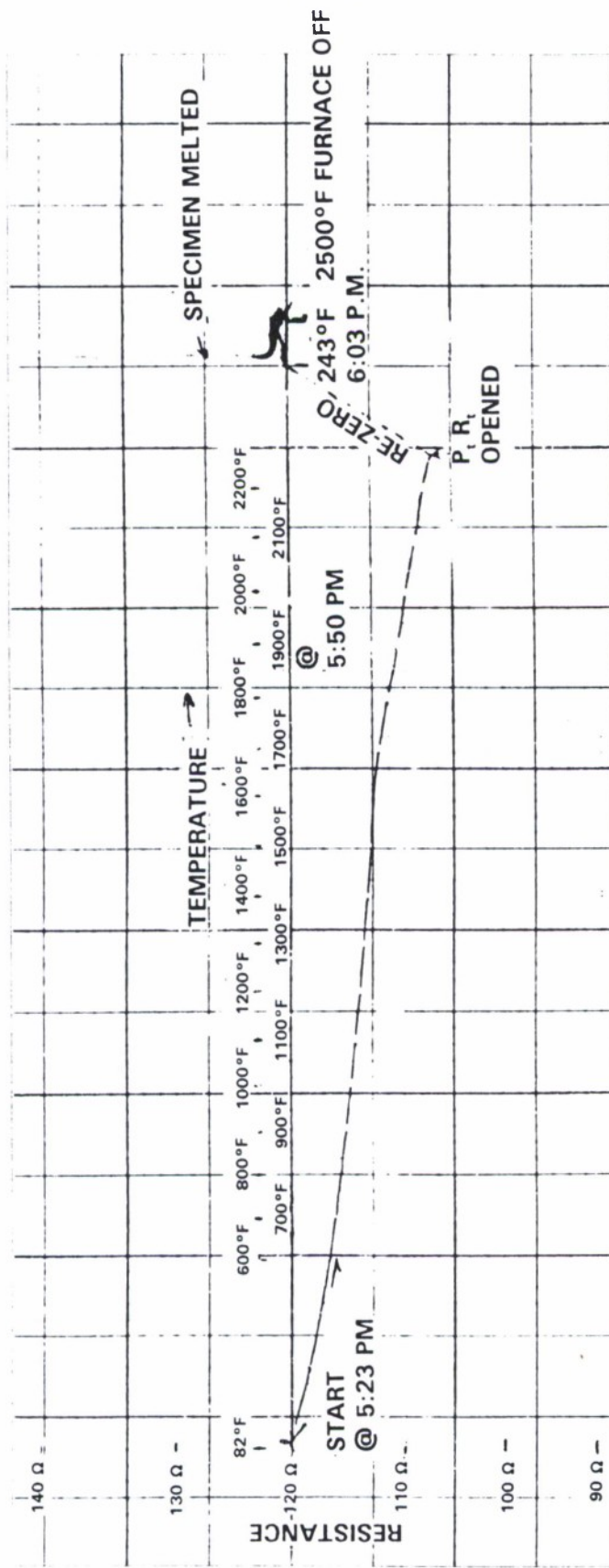


FIGURE 50. Resistance vs. Temperature

HDC Dual Element Strain Gage with Pt8W Sensor  
and ZGS Pt Thermometer

First Cycle Furnace Heatup to 2500°F

Hitec Products, Inc.

HDC-12-500-CPW2 on Rene' 41

Hitec FA Powder Flame Spray

$R_g = 120\Omega$  PT-8W 0.002" Dia. PtNi Leads,

Convolute & Flattened

$R_t = 25\Omega$  ZGS Pt 0.001" Dia. 0.003 PtNi Leads,

Convolute & Flattened

6VDC PWR.; Pt/Pt 10Rho. T.C.

$Y = 500 \text{ MV/}^\circ\text{F}$ ;  $X = 2 \text{ MV/}^\circ\text{F}$

3-3-90 SPW

second cycle. On the second heat up the gage opened at 2150°F and the test discontinued. See Figures 51, 52, and 53.

The third Rene' 41 specimen was the foil strain gage. See Figure 54. It was inserted into the furnace and recorded on the plotter up to 2423°F. See Figure 55. The specimen was stuck in the top opening and would not come out. Finally, part of the specimen melted and fell into the furnace. The furnace was turned off, the cover removed, and the remains of the specimen were retrieved with a pair of tongs.

#### 3.7.6.3 High Temperature Tests on 316 Stainless Steel

A Pt8W gage was installed on a 316 SS specimen with Rokide Hitec HT rod on to a no. 60 grit prepared surface. The 316 is an austenitic stainless having a higher thermal expansion coefficient than the Rene' 41 and a higher melting point (about 2550°F). The gage was tested using a slow heat up rate similar to the first Rene' 41 test above. Figure 57 shows the X-Y plot of the heat up and cool down cycle. The specimen had just begun to melt indicating a specimen temperature of 2550°F had been reached. See Figure 56.

#### 3.7.6.4 TZM Tests

The initial tests on TZM consisted of applying coatings of ceramic cement to a sandblasted surface and testing at 600°F, 800°F, and 1000°F. The cement coatings were 1) Yellow Cerro, 2) Brimor U529, and 3) Bean H. The specimen turned blue almost immediately at 650°F. However, each cement turned various colors not normal for the given cement.

The specimens were subjected to 24 hours at 800°F. The cements were still well bonded, but severely discolored. The furnace was turned up to 1000°F for three days. Upon removal, the specimen was severely oxidized, (Figure 59), and all cements were fractured and debonded.

A HBWАНV-12-125-.4MG gage<sup>27</sup> was spot welded to a TZM specimen and subjected to the same heat cycle as the specimen above. The weldable gage withstood the heat cycle well. See Figure 60.

An uncoated TZM specimen was inserted into a preheated furnace and subjected to 5 minute periods of progressively higher temperatures. At 1500°F the specimen began to "smoke" and grow large crystals as shown on the specimen in Figure 61. A second specimen was grit blasted and coated with patches of ceramic cement. Figure 62 shows 1) WC-9, 2) Red Cerro, 3) Yellow Cerro, and 4) Yellow Cerro. Exposure to 5 minutes at 1500°F resulted in a total loss of the cement due to flaking off or reaction with the substrate.



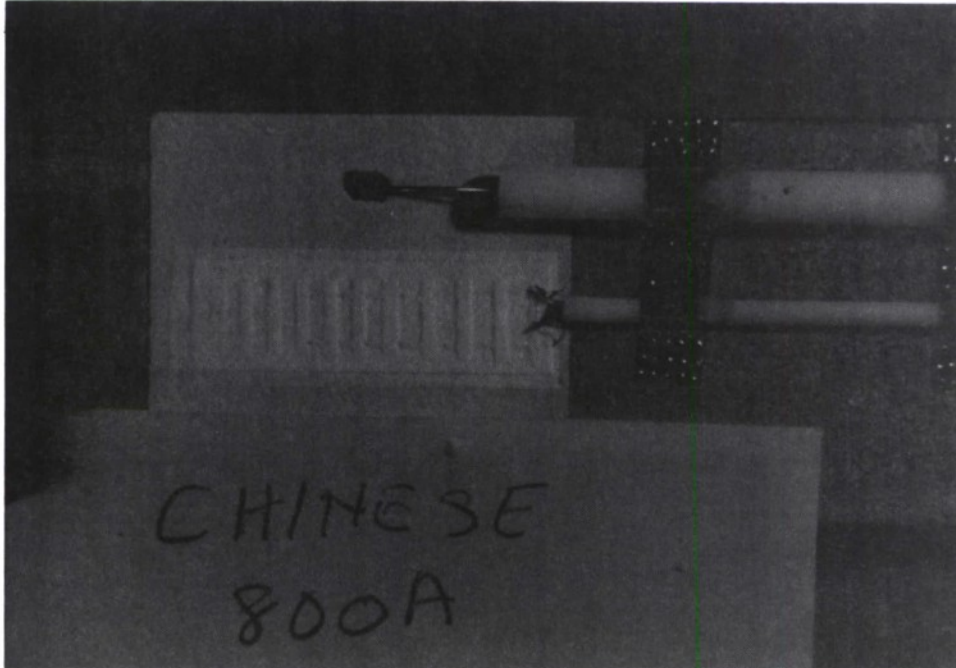


FIGURE 51. Chinese 800A Alloy Strain Gage Installed with FA Powder on Rene' 41 Bar

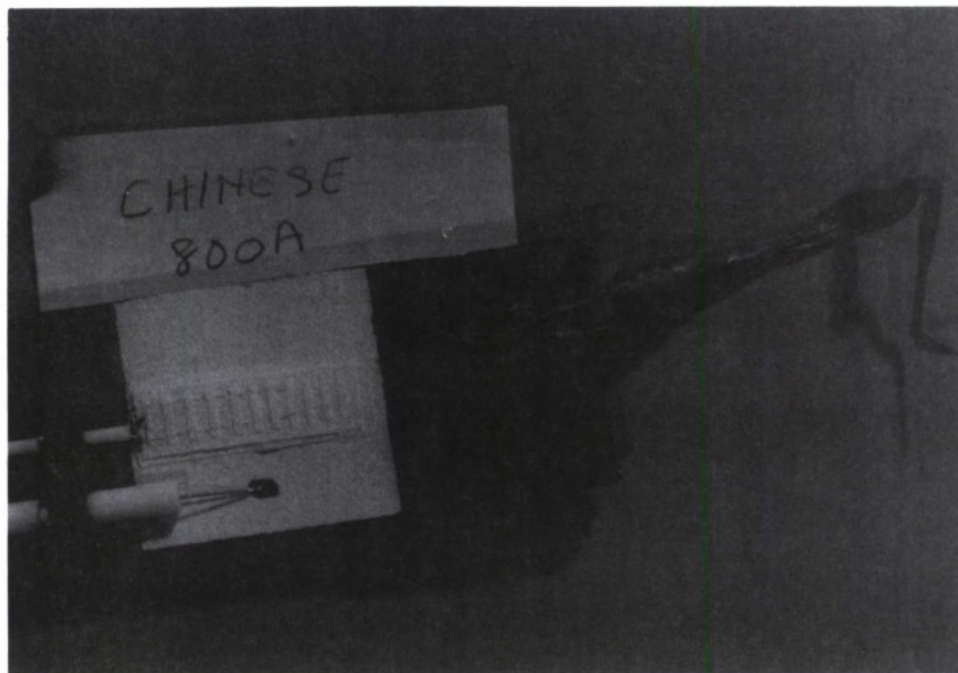


FIGURE 52. Chinese 800A Alloy Strain Gage After Second Cycle 2500°F

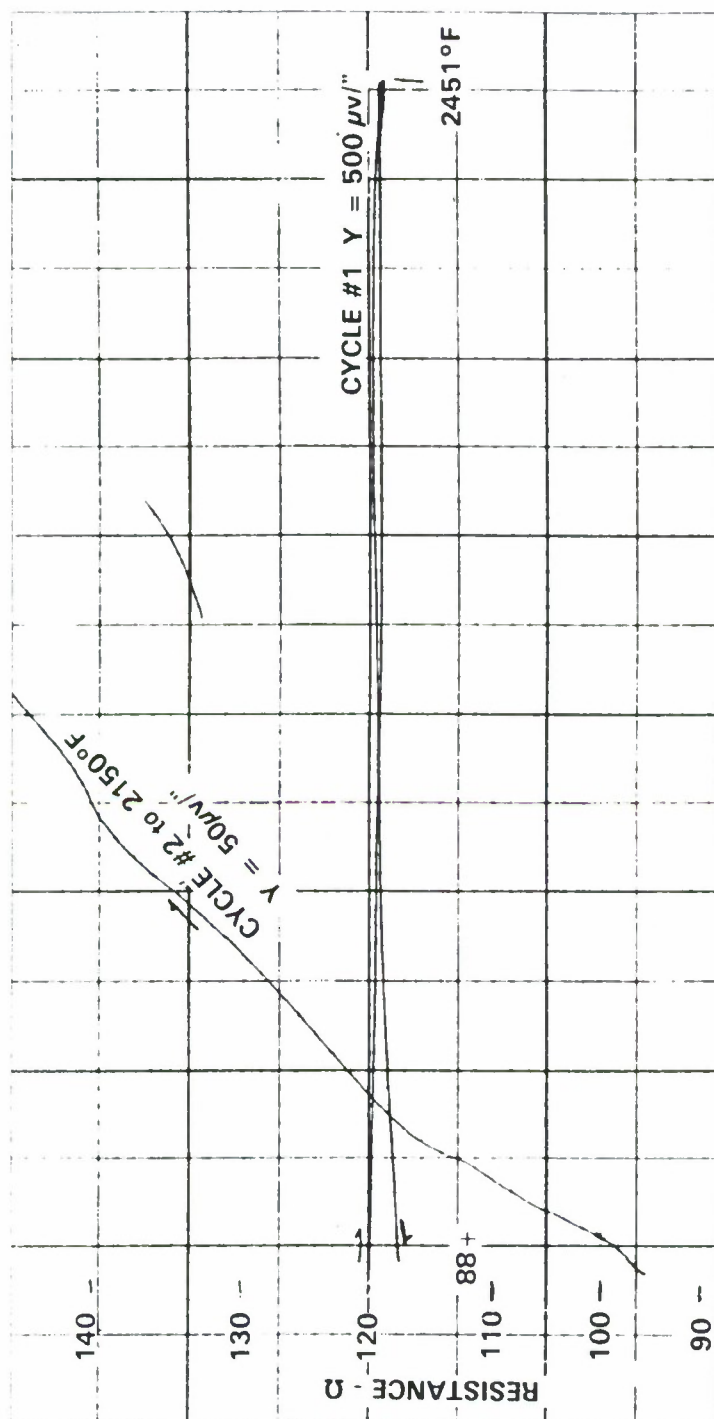


FIGURE 53. Resistance vs Temperature

Chinese 800A Alloy

Plunge into 2550°F Furnace

Specimen Melted on Second Cycle

Hitec Products, Inc.

HFC8-12-468-LCW on Rene' 41

Chinese 800A Alloy

Hitec FA Powder Flame Spray

$R_0 = 120.9\Omega$

6.0VDC PWR. Pt/Pr 10Rho. T.C.

3-3-90 SPW

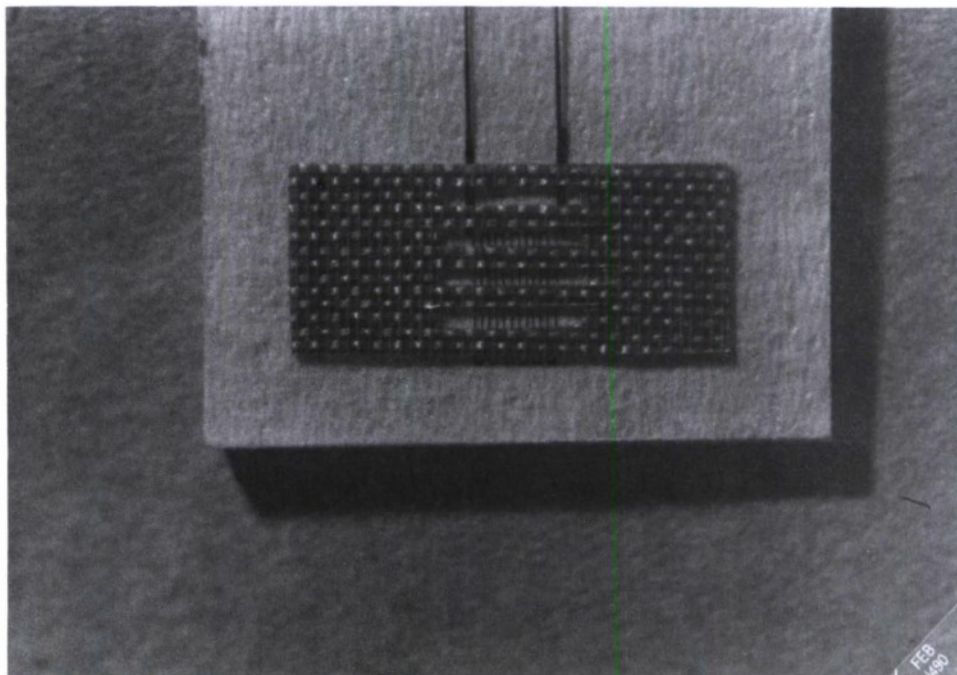


FIGURE 54. FeCrAl Foil Strain Gage Prepared for Installation by Powder Flame Spray. Note the large foil end tabs and lead pads have been cut off and a tape carrier applied. Leads are 5 mil chromel rolled flat in the weld area.



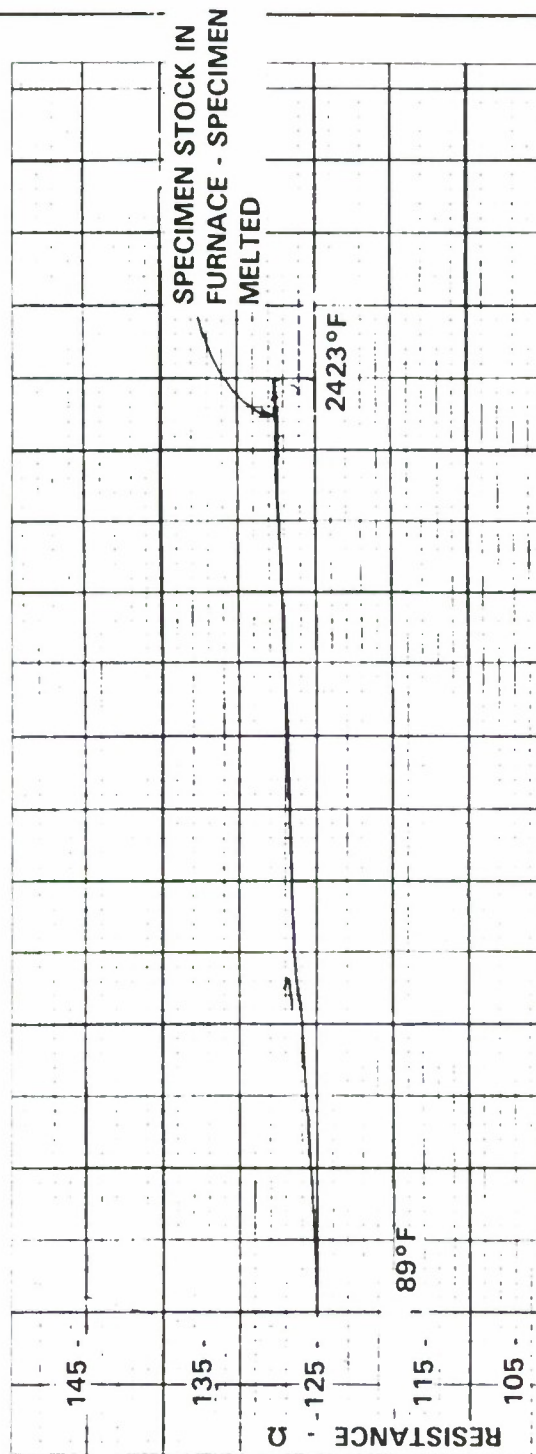


FIGURE 55. Resistance vs Temperature  
Plunge into 2550°F Furnace  
Specimen melted on first cycle.

Hitec Products, Inc.  
MEZ Type NZ 2104 Foil Gage on Rene' 41  
Hitec FA Powder Flame Spray  
 $R_g = 125.0 \Omega$   
6.0VDC PWR; Pt/Pt 10Rho. T.C.  
 $Y = 500 \text{ MV/}^\circ\text{F}; X = 2 \text{ MV/}^\circ\text{F}$   
3-3-90 SPW

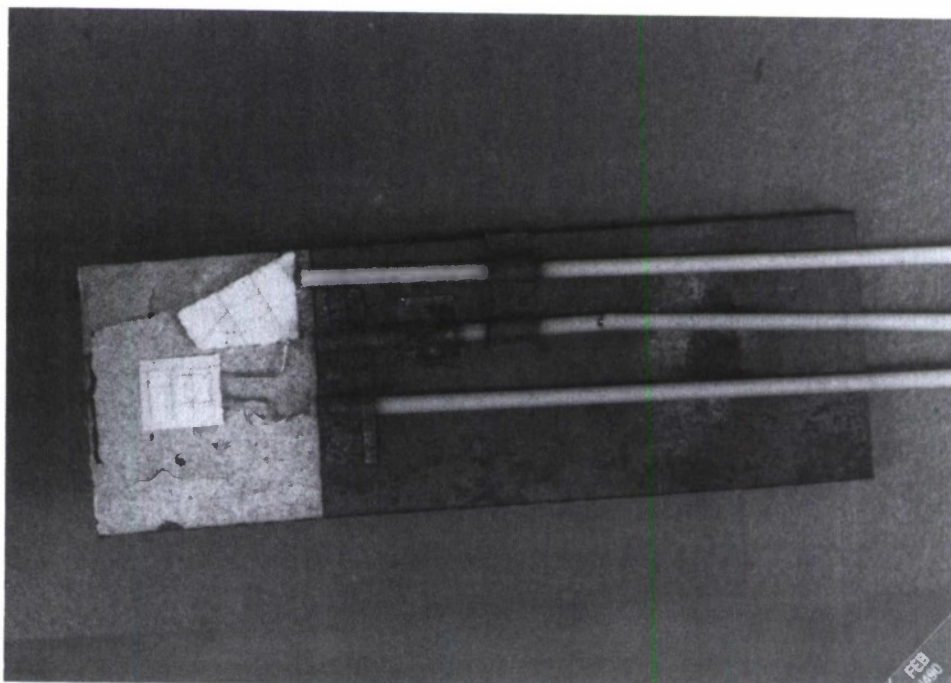


FIGURE 56. Pt8W Gage on 316 S.S. After Test to 2550°F.  
Note beginning of melting under alumina insulators near right  
end.

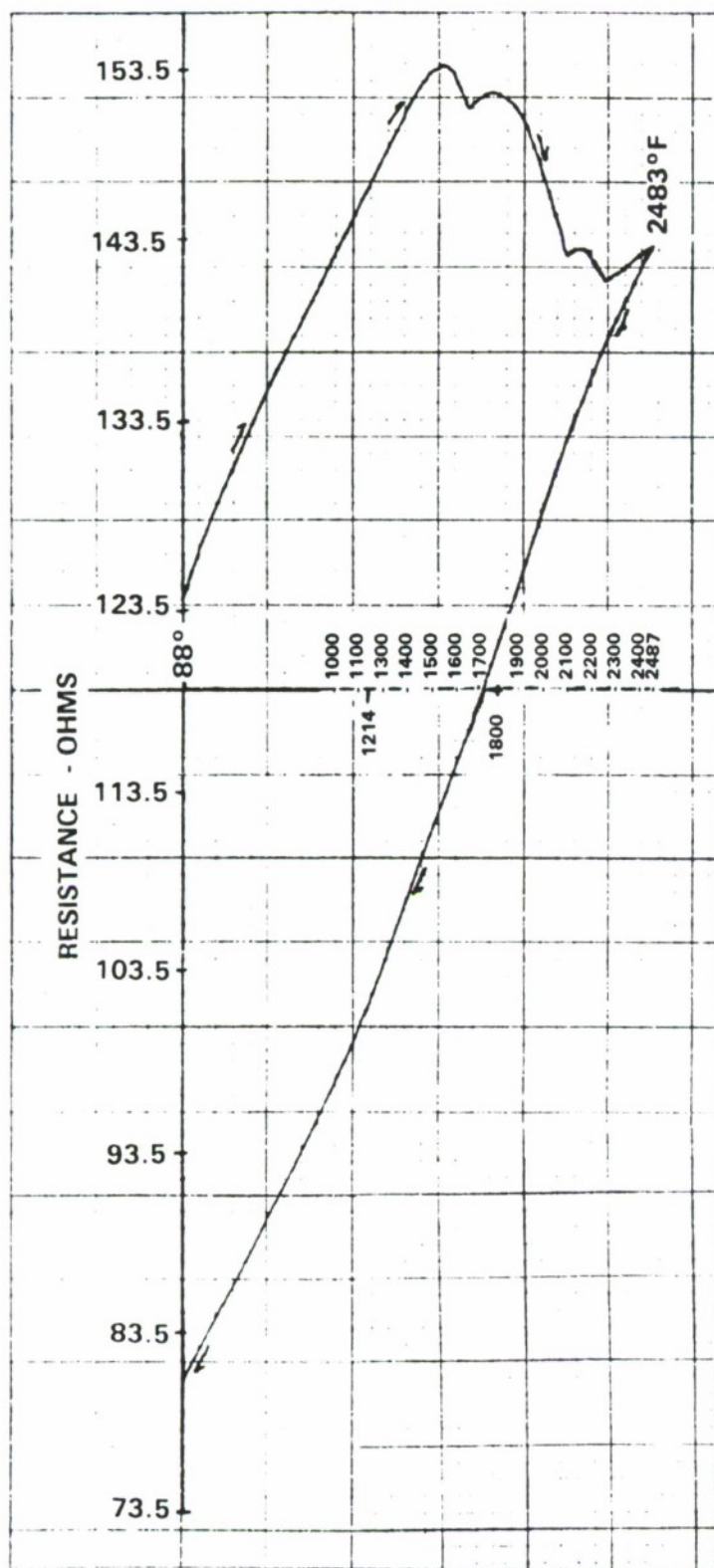


FIGURE 57. Resistance vs. Temperature  
Pt8W Alloy Strain Gage

Hitec Products, Inc.  
HFP-12-125-LPW on 316 S.S.  
Rokide Hitec HT Flame Spray  
Pt/Pt 10Rho. T.C.  
Start @ 6:55 p.m.  
2000°F @ 7:55 p.m.  
2400°F @ 8:23 p.m.  
2473°F @ 8:33 p.m.  
2483°F @ 8:35 p.m.  
Specimen Partially Melted  
Melt Point 2550°F  
1-11-90 SPW



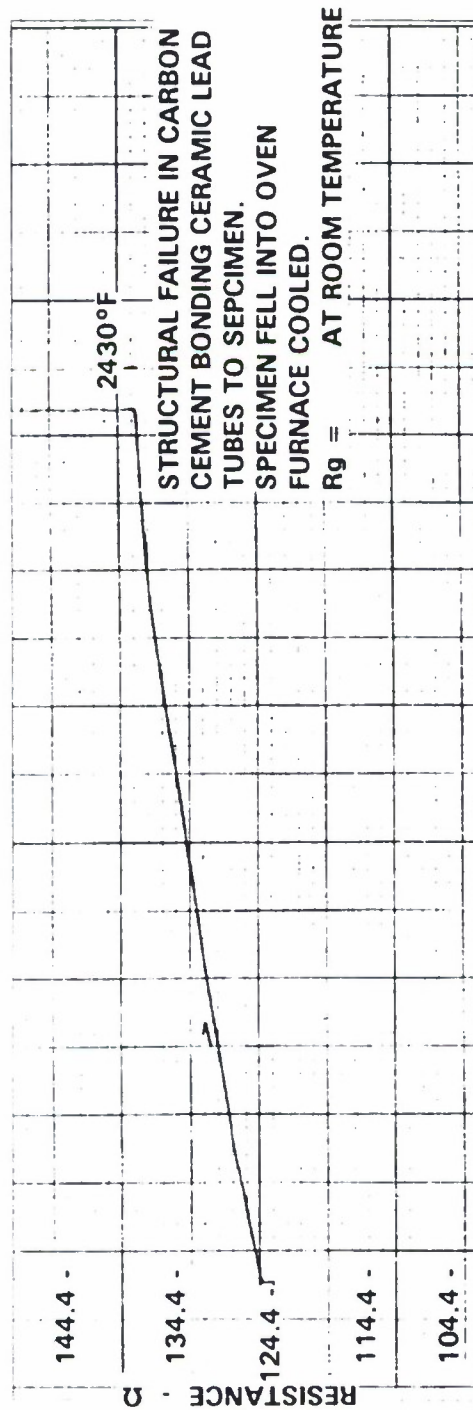


FIGURE 58. Resistance vs. Temperature  
Pt8W Strain Gage on C/C

Hitec Products, Inc.  
HFP-12-500-CPW2  
On Thiokol Carbon Composite  
Hitec FA Powder Flame Spray  
 $R_g = 1.24 \Omega$   
6.0 VDC BRG. PWR.

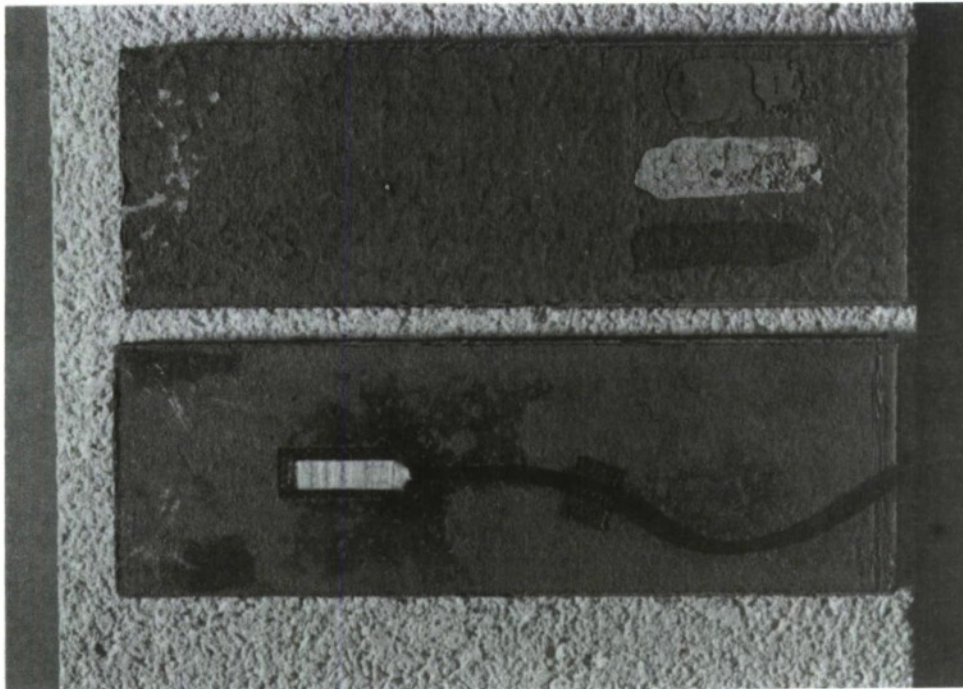


FIGURE 59. (Top Bar) Ceramic Cement Precoat on TZM After Exposure to 1000°F for 3 Days

FIGURE 60. (Bottom Bar) Weldable Strain Gage Installed on TZM by Spot Welding and Exposed to 1000°F for 3 Days

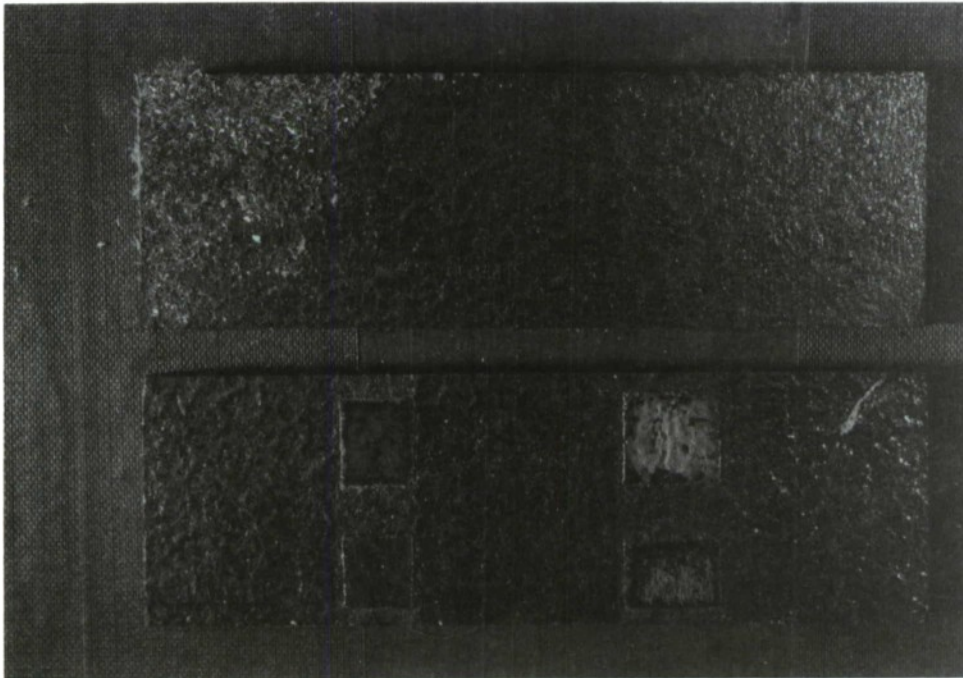


FIGURE 61. (Top Bar) An uncoated TZM specimen begins to "smoke" at 1500°F and grow large crystals on the surface.

FIGURE 62. (Bottom Bar) TZM Specimen with Precoat Patches of Ceramic Cement. Counter Clockwise From Top Left:  
1) Yellow Cerro, 2) Yellow Cerro, 3) WC-9, and 4) Red Cerro

Since the TZM specimens were extremely large, it was decided to experiment with pure molybdenum specimens 20 mils thick and 1/2 inch x 1 1/2 inches. Sodium silicate coatings, and SC cement coatings were tried as well as Rokide A over a grit blasted surface. Rokide impregnated with sodium silicate protected well to 2600°F. Total coating thickness was 4-6 mils. Standard FA powder gage installations using the CR-1000 Mini-Gun bonded well to this coating. Pt8W gages and type S thermocouples were applied to the coated specimens. Ten mil platinum leads were fed through alumina two hole insulators and spot welded to the gage leads. The alumina insulators were bonded to the specimen using WC-9 cement and cured 1/2 hour at 200°F, 1/2 hour at 400°F, and 1 hour at 650°F. The Pt8W gage was tested for 6 cycles to 2550°F. Although some intermittent shorting of the leads occurred, the gage and thermocouple were still working after the sixth cycle.



#### 4.0 CONCLUSIONS

The following were accomplished during this program:

1. Compilation and tabulation of strain gage types, adhesives, lead wires and attachment techniques for carbon composites and Rene' 41 for testing in the 600°F to 2500°F temperature range.
2. Development, evaluation and use of a pull tester to screen strain gage adhesives was completed.
3. Tests indicating severe reactions between strain gage ceramic cements and TZM precluded their use above 1400°F (short term), and 800°F (long term). A silicate impregnated Rokide flame sprayed alumina coating was developed to permit short term testing of TZM in air up to 2600°F.
4. Significant refinements in flame spraying processes to include vortex type air cooling, gage installation and spraying technique provided strain gage installations with the best electrical properties and bond strengths capable of withstanding repeated furnace plunges to 2550°F.
5. A prototype low profile GPD capacitive strain gage developed under NASA contract was successfully tested for apparent strain to 2000°F.
6. WC-9 experimental ceramic cement was successful in bonding thermocouples to carbon composites to 2550°F and for bonding terminals and insulators to carbon composites and alumina coated TZM specimens.
7. FeCrAl alloy foil (ME-2 2104) gages were successfully bonded to Rene' 41 using flame spray techniques and tested to 2423°F.
8. Chinese 800A alloy strain gages were also successfully flame spray bonded to Rene' 41 and tested to 2451°F.
9. Platinum tungsten strain gages were successfully flame spray bonded to Rene' 41, carbon/carbon and TZM and successfully tested to 2550°F.
10. Silicon carbide coated carbon composite, precoated with experimental SC cement, and instrumented with PT-W strain gage and type S thermocouple, successfully withstood 2 cycles to 2500°F without cement failure.
11. Installation techniques for ceramic cement and flame spray are fully documented and appended to this report.

## 5.0 LIST OF REFERENCES

1. Lei, Jih Fen, "PdCr Strain Gages Measure Static Strain at High Temperature." Advanced Materials and Processes, May 1992
2. Lanius, S.J., and Brasfield, R.G. "Final Technical Report for the SRM Nozzle Instrumentation and Model Validation Study." Vols. 1 & 2, Contract No. NAS8-36290, Morton Thiokol Inc., P.O. Box 241, Elkton, MD
3. Wnuk, S.P. Jr. "Current Developments in High Temperature Strain Gages." VI International Congress on Experimental Mechanics, Portland, OR, June 5-10, 1988
4. Howard, J.L., Nelson, E.J., and Barthel, C.A. "Recent Developments in High Temperature Capacitance Strain Gage Technology." Proceedings Congerence on Experimental Mechanics, Cambridge, MA, SEM. Bethel, CT, 1989
5. LEX-10. Product Data Sheet, Hitec Products, Inc., Ayer, MA 01432
6. SC Cement. Product Data Sheet, Hitec Products, Inc. Ayer, MA 01432
7. Hitec Mini-Gun Flame Spray System. Product Data, Hitec Products, Inc. Ayer, MA 01432
8. Moore D.G., "Bonding Agents for High Temperature Strain Gages." STP-230 ASTM etc., Pgs. 106,107
9. Pitts et al., "Ceramic Type Gages." STP-230, ASTM (Average resistivity and strengths of propriety and experimental cements)
10. Sermatech, "Surface Engineering Bulletin no. 200." (Contains various properties of strain gage cements), Sermatech International Inc., Limerick Road, Limerick, PA 19468
11. Rocal Ltd., "Brimor High Temperature Cements." Technical data sheet no. 900, Rocal House, Swillington, Leeds, LS26 8BS U.K.
12. Barthel C.A, Howard J.L, Nelson E.J, Mullineau J.L., "High Temperature Measurements on Uncoated Carbon/Carbon material," Experimental Techniques, Pg. 33-38, September 90
13. Leszynski S., "Flame Sprayed Sensor." Proceedings WRSGC, Pg 14-29, (Ref. on powder flame spraying, good test fixture), October 1961

14. Wnuk S.P., "Development of Strain Gages for Vibratory Strain Measurements on Advanced Gas Turbines." TCSG Proceedings, Cleveland, 1982
15. Wnuk S.P., "Progress in High Temperature and Radiation-Resistant Strain Gage Development." (General background on leakage to ground and electrical properties of ceramics). Experimental Mechanics, Pg. 27A - 33A, May 1965
16. Wood S., "A Study of Resistance to Ground Properties of Ceramic Adhesives at High Temperatures." Sixth Hostile Environments Conference, Kansas City, Nov. 89, SEM, Bethel, CT
17. Anon. "A Megohmmeter Circuit for SR-4 Strain Indicators." BLH Measurement Topics, BLH Electronics, Canton, MA USA, June 1966
18. Kottkamp E, Wilhelm H., and Kohl D., "Strain Gauge Measurements on Aircraft." AGARD graph No.160, AGARD flight test instrumentation Series, Volume 7, NATO Advisory Group for Aerospace Research & Development, 7 Rue Ancella Neuilly Sur, Seine France
19. Drumheller C.E., "Study of Internal Static Friction in Plastic Bonding Materials for Resistance-Wire Strain Gages." Doctoral Thesis, Pennsylvania State College, January 1953
20. Stein P.K., "Stresses Within the Adhesive Layer of a Strain Gage Installation." (Taken from Analyse Des Constraints, Volume II, by G. Fouretier, Strain Gage Reading References 9-817)
21. BLH Lab Notes, "Theoretical Analysis of Shear Stresses and Forces Within a Strain Gage Wire Imbedded in Adhesive." Anon., Circa 1962
22. Sharpe L.H., Schonhorn H, Lynch C.J., "Adhesives." International Science and Technology, April 1964
23. Hudson, J.M., "Strain Gage Heating During Flame Spray Application." Report No. R69AEG162, General Electric Aircraft Engine Group, Evandale, OH
24. Wnuk, S.P. Jr., Low, M.E., and MacLean, J.G. "Development of Strain and Temperature Measurement Techniques for Use in Combined Thermal Acoustic Environment." AD B000924-4 NTIS, 5285 Prot Royal Road, Springfield, VA 22160, October 1974
25. Wnuk, S.P. Jr., Wnuk, S.P. III, and Wnuk V.P. "High Temperature Capacitive Strain Gage." NASA Contractor Report 4266, Contract NAS1-18668, NASA Langley Research Center, VA



26. Ma, L., Wu, T.T., and Ahzo, L. "Development of Temperature Compensated Resistance Strain Gages for Use to 800°C." Proceedings of the International conference on Experimental Mechanics, Beijing, Peoples Republic of China. Science Press, Beijing, October 1985
27. Weldable Strain Gages. Product Data Sheet, Hitec Products, Inc., Ayer MA 01432
28. Evans, H.E., "Strain Sensor for High Temperatures." U.S. Patent 3,534,479, 20 Oct., 1970
29. Vranas, T., "High Temperature Strain Gage Calibration Fixture." US Patent 3,956,919, 18 May, 1976
30. Bunshah, R.F., "Two Vapor Coating Processes Cover The Field of Applications." (CVD Techniques), Research & Development, pgs. 73-78, August 1989
31. Jacques, H., "Platinum Temperature Sensors: A New Generation." (Pt. thin film resistors laser cut, usable to 600°C), Sensors, May 1989, pgs. 99-109
32. Horton, L.L., "In Search of New Materials." (Nickel aluminides, iron-chromium-nickel alloys), Mechanical Engineering, September 1988 Pgs. 44-49
33. Smith, D.K., "Diamond Thin Film Deposition Source." Research and Development, pg. 92, October 1988
34. Orback, H.K., "How Ceramics Use Plasma Jet." Ceramic Industry, November 1962
35. Gardner, A.R., "New Paints and Cements Make Conductive Coatings." (Gold, silver, carbon and platinum thick film coatings usable to 1700°F), Product Engineering, February 18, 1963
36. Stephen R.I., Sinclair G.M., "A High Temperature, Vacuum, Axial Fatigue Testing Machine." (2500°F, excellent design), ASTM Bulletin, Pgs. 44-48, October 1960
37. Fisher R.H. , Carlson R.L., & Holden F.C., "Apparatus for Mechanical Testing at Temperature Above 2000°F." (Actually 2000 to 4000°F), Materials Research & Standards, January 1962
38. Jaffe D.L., Rittenhouse J.B., "How Materials Behave in Space." Materials in Design Engineering, September 1962
39. Riley, M.W., "Carbon and Graphite." Manual No. 199, Materials in Design Engineering, September 1962

40. Peckner, D., "Refractory Metals." Special Report No. 200, Materials in Design Engineering, (Properties of TZM, Niobium, Tantalum and Tungsten) October 1962
41. ASTM, "Standard Test Method for Adhesion or Cohesive Strength of Flame Sprayed Coatings." ASTM: C633-79, American Society for Testing and Materials, 1916 Race St. Philadelphia, PA 1910B, Rev. July 1985
42. Quad Group, "Coating Tests" (Bond Strength, Pin Pull Test, Thin Film, Screen or Plated Coatings Tests) Quad Group, S.1815 Lewis, Spokane, WA 99204
43. ETP, "Hybrid Microelectronic Test & Production Equipment." (Pull Test Equipment), ETP Engineered Technical Products, Inc., 3380 US H'way 22, Summerville, NJ 08876
44. Mecmesin, Inc., "Force Measurement Fixturing and Accessories." (Pull test machines and accessories for testing electronic components, wire, crimped wire, terminals, lugs etc.) Mecmesin Inc., Lawson Hunt Industrial Park, Broadbridge Heath, West Sussex RH12 3JR, England
45. Alessi, "Failure Analysis Test Station." (Micro manipulator and probes for probing and testing of IC under the microscope). Alessi, Inc., 16871 Noyles Ave., Irving, CA 92714
46. Littleton H.E, Pears C.D., "Tensile Properties of High Modules Fibers." (A miniature tester for testing single filaments of tungsten, molybdenum, carbon, boron and steel fibers). Research Development, June 1966
47. Deutchman, A.H., "Ion Implantation and Ion Assisted Coatings - Improved Bond to Metals." Private Communication, Beamalloy Corp., 6360 Dublin Industrial Park, Dublin, OH 43017, October 1990
48. Curie, L.M., Hamister V.C. & MacPherson H.G., "The Production and Properties of Graphite for Reactors." National Carbon Co., Division of Union Carbide Corp., 30 East 42nd St., New York, NY
49. Bonn J.C., "Wire Grid Forming Apparatus." US patent 2,991,671, July 1961
50. Cotronics Corp, "Ceramics and High Temperature Materials Handbook." Cotronics Corp., 3379 Shore Parkway, Brooklyn, NY 11235
51. JP Tech, "Installation Summary, High Temperature Foil Gages and Bean H Cement." JP Technologies, 42 N. Benson Ave, Upland, CA 91786

52. Hartman G.A., Zawada L.P. & Russ S.M., "Techniques for Elevated Temperature Testing of Advanced Ceramic Composites." Hostile Environments and High Temperature Measurements Conference, Costa Mesa, CA, SEM, Pg. 31-38, March 1988
53. Hulse C., Bailey R., "Extreme Temperature Strain Measurement System." Final Report, Contract WRDC-TR-90-3054, Flight Dynamics Laboratory WRDC, Wright Patterson AFB, OH, August 1990
54. Lei J, Williams W., "PdCr Based High Temperature Static Strain Gage." AIAA Second International Aerospace Planes Conference, Oct. 1990, AIAA 370 L'Enfant Promenade, SW, Washington DC 20024
55. Sigmond Cohn Corp., "Platinum and Platinum Alloys." Sigmond Cohn Corp., 121 So. Columbus Ave, Mt. Vernon, NY 10553
56. Sigmond Cohn Corp., "Properties of Metals and Alloys." Sigmond Cohn Corp., 121 So. Columbus Ave, Mt. Vernon, NY 10553
57. Hoskins Mfg. Co., "Physical Properties of Chromel." Hoskins Mfg. Co., 10 776 Hall Road, Hamburg, MI 48189
58. Driver Harris, "Designing Heating Elements." Driver Harris Co., Harrison, NJ 07029
59. Kanthal Corp, "Electrical Properties of Electronic Alloys." The Kanthal Corp., Wooster St, Bethel, CT 06801
60. Whuk S.P., "Attachment Techniques for High Temperature Strain." Phase I final report, Flight Dynamics Laboratory, WRDC/FIBTA Contract No. F33615-89-C-3211, Wright Patterson AFB, OH, 45433, March 1990
61. Fullam, E.A., "Accessories for Microscopy." (Mechanisms for tensile testing, 3 and 4 point bending in vacuum under a scanning electro microscope), E.J. Fullam Inc., 900 Albany Shaker Road, Latham NY 12110.
62. Zhu Yon-Liang, "Self-Compensated High Plastic Resistance Strain Gauges For Use Up To 300°C." (High elongation lead design), Strain, Pgs. 181-185, November 1987
63. Bertodo, R., "Platinum Metal Alloys for Measurements of Steady Strain at High Temperature." Proc. Inst. Mech. Engrg., Vol 178, No.34, pg. 907, 1964
64. Bertodo, R., "Precious Metal Alloys for High Temperature Resistance Strain Gauges." Brit. Jour. Appl. Physics, Ser 2, Vol 1, Pg. 1743, 1968



65. MTS, "Silicon Carbide 4 Point Bending Fixture." MTS System Corp., P.O. Box 24012, Minneapolis, MN 55424
66. Instron, "High Temperature Ceramic Test fixtures." Instron Corp., 100 Royal St, Canton, MA 02021
67. Hulse C.O, Bailey R.S., Grant H.P. Przybyszewski J.S., "High Temperature Static Strain Gage Development Contract." Contract No. NAS 3-23722, NASA Lewis Research Center, Cleveland, OH, 1985
68. Norton, "Model 201 Igniter Assembly (Silicone Carbide)." Norton Co., Worcester, MA 01606, September 13, 1983
69. Struble, L., "The Performance of Portland Cement." ASTM Standardization News, January 1992
70. Nekhendzy, E. Ju., Private Communication, Polzunov Boiler and Turbine Institute, Leningrad, Russia.
71. Balaguru, P.N. Shah, S.P., "Fiber Reinforced Cement Composites." McGraw-Hill Inc., New York, NY, 1992
72. Faires V.M., "Design of Machine Elements", The MacMillian Co., New York, 1955
73. Dykes, Burke C., "Strain Measurements at Temperatures From 500 to 1800°F." ISA Preprint, Number 16., September 1, 1966
74. Anonymous, "Rokide Training Course." Hitec Products Inc., Ayer, MA 01432
75. Francis, A. and Haynes, K., "Apparent Strains in Unmounted Strain Gauges at High Temperature Strain." August 1991
76. Wnuk, S.P., "Performance Characteristics of Bonded Strain Gages Under Repeated Cyclic Strain." Presented at SEM meeting, Cleveland, OH, October 1964
77. Dorsey, J., "New Developments and Strain Gage Progress." Proceedings of Western Regional Strain Gage Committee, Fall 1965
78. Bertodo, R., "Measurement of Steady Strains at Temperatures Above 650°C." Journal of Strain Analysis, Vol. 1, No. 1, pg. 15, 1965

79. Wnuk, S.P., "Innovations in Flame Spraying Techniques." Proceedings of the Regional Strain Gage Committee, Albuquerque, NM, April 1965
80. Lei, Jih-Fen. "Development and Characterization of a PdCr Temperature Compensated Wire Resistance Strain Gauge." Fourth International Conference of Machine perception Technology, Cleveland, OH, Expocon, Trumbull, CT, September 1989
81. Grant, H.P., claing, R.G., Mazur, H.J., and Zambrano, C.A. "Test of PdCr and BCL-3 Wire Resistive Static Strain Gages to 1600°F." Sixth Hostile Environments and High Temperature Measurements Conference, Kansas City, SEM, Bethel, CT, November, 1989
82. Hulse, C.O., Bailey, R.S., and Lempkey, F.D. "High Temperature Static Strain Gage Alloy Development Program." Contract No. NAS3-23169, NASA Lewis Research Center, Cleveland, OH, 1985
83. Wnuk, S.P. Jr. "High Temperature Strain Gage Alloys." International Conference on Experimental Mechanics. Beijing, Peoples Republic of China, Science Press, Beijing, China, 1985
84. Harting, D.R. "Evaluation of a Capacitive Strain Measureing System for Use to 1500°F." ISA ASI 75251 (289-297), 1975
85. Lemcoe, M.M. "High Temperature Strain Gauge Measurements to 2000°F. Can it be Done?" Fourth International Conference of Machine Perception Technology. Cleveland, OH, Expocon, Trumbull, CT, September 1989
86. Kershisnik, D.P. "High Temperature Strain Gage Techniques on Graphite." 1973 Spring Meeting, Los Angeles, SEM, Bethel, CT, May 1973
87. Micro Engineering II. Catalog, Upland, CA
88. Anon. Product Data Bulletin. Hitec Products, Inc., Ayer, MA 01432
89. Strain Gage Catalog. Eaton Corp, Troy, MI
90. HDA/HDB Strain Gage. Installation Instructions, Hitec Products, Inc., Ayer, MA 01432
91. Wnuk, S.P. Jr. "High Temperature Strain Gages for Short Term Static Testing." Proceedings T.C.S.G. SEM, Brookfield, CT, May 1974

# APPENDIX A

## BONDING FREE FILAMENT STRAIN GAGES WITH CERAMIC CEMENT

- I. GENERAL
- II. FACILITY REQUIREMENTS
  - A. Tools
  - B. Materials
- III. SAFETY PROCEDURES
- IV. SURFACE PREPARATION
  - A. Grit Blasting
  - B. Nickel Aluminide
- V. GAGE APPLICATION
  - A. Masking
  - B. Cement Precoat Application
  - C. Cure Cement Precoat
  - D. Apply Gage
  - E. Apply Cement
  - F. Cure Cement RT + 1/2 Hour 200°F 5-10 Minutes @ 350°F
  - G. Remove Temporary Tape Carrier
  - H. Bake at 800°F
    - I. Apply Final Coating
  - J. Final Cure
- VI. WATERPROOFING
- VII. QUALITY CONTROL MEASUREMENTS AND RECORD KEEPING



## **I. GENERAL**

These procedures apply for the following ceramic cements:

Yellow Cerro, Hitec 710, Brimor, Chinese P12-9, PBX, GA-100, Bean H.

## **II. FACILITY REQUIREMENTS**

A clean, humidity controlled, quiet, well lighted room with a bench and a chair, a 7x to 30x wide field stereo zoom microscope with light, a furnace or heat source to cure ceramic cement to 800°F are necessary. Humidity should not exceed 47% R.H.. Either a grit blast cabinet or flame spray equipment set up to apply metal coatings is required for surface preparation.

### **A. Tools**

The following tools are required:

- 1) Spatula, stainless steel,
- 2) 6 inch machinist's rule,
- 3) Metal scribe,
- 4) Pick,
- 5) Sable artist's brushes, 10/0, 6/0, 4/0, no. 1, 00,
- 6) Wiss embroidery scissors,
- 7) Tweezers Dumont, no. 3C, Rio Grande Style 5,
- 8) 2 inches x 6 inches glass sheet,
- 9) Industrial razors, single edge,
- 10) Carbide tip scribe,
- 11) Colored pencil, red,
- 12) X-acto knife,
- 13) Polypropylene sponge brush, 1 inch wide
- 14) Digital volt-ohm meter

### **B. Materials**

- 1) Transparent tape, ½ inch wide,
- 2) Fiberglass reinforced teflon tape, 1 inch to 1 ½ inches wide,
- 3) Aluminum teflon tape, ½ inch wide,
- 4) Masking tape, 1 inch wide,
- 5) Grit blasting, tape 1 inch wide,
- 6) 120 grit, aluminum oxide grit,
- 7) No. 120 grit, abrasive cloth,
- 8) Gauze pads, 2 inches x 2 inches

- 9) "Q" tips (wooden),
- 10) Alcohol, isopropyl,
- 11) Methyl ethyl ketone (MEK), safety bottle,
- 12) Ceramic cement,
- 13) Surgical gloves,
- 14) Cotton protective clothing.

### **III. SAFETY**

Cleaners, solvents, tapes containing teflon, and ceramic cements contain toxic and/or hazardous materials. Be sure to read the M.S.D.S sheets and comply with all precautions and handling recommendations.

### **IV. SURFACE PREPARATION**

Remove all dirt, grease, paint, scale and oils, using appropriate tools and solvents. Test specimen may also contain microcracks or porosity which contain contaminants that are not removed by surface cleaning. Rolled sheet and especially titanium alloys contain embedded contaminants which can only be eliminated by baking at 800°F for about 2 hours. After gross contaminants have been removed, and the specimen baked at 800°F, prepare the surface for cement bonding as described below.

#### **A. Grit Blasting**

In order to achieve good cement bonding, the surface must be clean and roughened using grit blasting, abrasive cloth roughening, or flame spray application (2 to 3 mils thick) of nickel aluminide. The choice of procedure depends on the part being bonded. Grit blasting can introduce residual stresses into the part which may be detrimental. Thin material may distort, warp, curl or pucker due to residual stresses. Blasting at an angle of about 30 degrees from the horizontal will usually introduce the least stress into the structure. This type of surface preparation requires a fully-developed technique by the operator.

#### **B. Nickel Aluminide**

The best surface preparation for most metals is achieved by applying a thin flame sprayed coating of nickel aluminide. The surface texture is very similar to a grit blasted surface, and if the thickness is kept to a minimum, no distortion due to residual stresses will occur. The key is to stop the coating process when the surface is about 95% covered. This results in a coating about 2-3 mils thick. Thick coatings can result in part distortion.

## **V. GAGE APPLICATION**

### **A. Masking**

The perimeter of the bonding area for the gage and lead wire should be masked off using appropriate masking tape. For grit blasting, use masking tape or other suitable grit blasting tape to outline the gage and lead bonding path. A path about ½ inch wide is typical. Protect all other areas of the part by shielding it with metal foil, sheet metal, or masking tape. For a nickel aluminide flame sprayed coating, masking should be done using teflon-fiberglass tape with silicon adhesive or teflon-aluminum tape with silicon adhesive. All other areas should be shielded from flame spray using appropriate shielding materials as for grit blasting. After surface preparation is complete, remove all shielding and masking materials. Be sure to remove all adhesive residue at the edge of the tape. Pick off larger pieces of adhesive residue with tweezers. Remove remainder of adhesive using a cotton "Q" tip dipped in MEK. Swab away from the bond area. Do not drag adhesive residue into the bonding area with the "Q" tip. There may be particles of grit embedded into the surface. These may be removed by passing the sharp side of an industrial razor over the surface, holding the blade at 90 degrees to the surface. Discard the razor after use. The same procedure as above is also used for a nickel aluminide precoat, including the razor treatment. Blow off any remaining particles using a blast of clean air. Be sure to wear eye protection with side shields when cleaning with air.

### **B. Cement Precoat Application**

Mask the perimeter of the area to be precoated with cement using transparent tape. This tape is about 1 ½ mil thick and is perfect for setting the precoat thickness.

Tools required: spatula, razor, gauze pads, no. 1 artist brush or polypropylene sponge paint applicator, ceramic cement. Be sure to have surgical gloves on when working with or cleaning the polypropylene brush.

Thoroughly mix the ceramic cement with the spatula until a uniform consistency is achieved. Some cements may require the use of a screwdriver to start the stirring process, especially if the cement has been sitting on the shelf for a while. Using the no. 1 brush (if the precoat area is small) dip brush into cement and wipe off with gauze pad several times until brush readily picks up cement. Gauze pad should be held in one hand. Protect hand with a surgical glove. Keep cement thoroughly stirred with spatula. Pick up a fully loaded brush and apply cement precoat quickly working the brush over the surface, wetting the surface as fast as possible. The cement dries rather quickly on the brush. As soon as fluidity ceases, wipe remainder of cement off brush into the gauze pad and reload brush with fresh well mixed cement. Once the entire area is covered, take a fully loaded brush and apply wet cement into the partially drying initial material, and working quickly with the brush, smooth out the precoat as uniformly as possible. While the cement is very wet, scrape the surface level to the thickness of the perimeter



tape by passing a razor or spatula blade over the wet cement. Push excess cement off the far end of tape and remove this excess using the gauze pad. Use the gauze pad to wipe off the razor, spatula, and the tape perimeter. The gauze pad is also used to wipe cement off cap and neck of bottle. The cap and interior bottle neck must be kept free of cement, otherwise the liquid will migrate out of the bottle, no matter how tightly the cap is screwed on. Keep cap on when not in use to prevent evaporation of liquid component.

Air dry precoat for at least ½ hour at room temperature. Drying time depends on relative humidity. In the winter with R.H. equaling 20% or less, ½ hour drying time is adequate. If R.H. is 47%, 6 or 8 hours of drying time may be needed. After air drying, remove all transparent tape. This tape must be removed prior to the cure or a gooey mess will result.

### **C. Cure Cement Precoat**

Cure precoat for one-half to one hour at 200°F followed by 1/2 to 1 hour at 350°F to 400°F. Cool to room temperature. At 200°F the cement gives off chemically bound water and turns hard. It may appear quite hard after the heating process. However, if allowed to remain at room temperature for a long time, moisture will be reabsorbed into the cement and it will again become soft. A 350°F cure is necessary in order for the cement to remain hard.

If large areas require precoating, a polypropylene sponge brush may be used to apply cement instead of the artist's brush. It may be necessary to thin the cement by adding 10-20 drops of thinner and mixing cement thoroughly. Dip the brush into the cement. Squeeze out the cement on the inside of the bottle and reload sponge. Note while doing this how well the cement wets the sponge. It is usually necessary to squeeze out the cement several times before the cement readily wets the sponge. When the cement wets the brush, dip the brush into fully mixed cement and squeeze most of the cement out, then apply cement to precoat area and work quickly into surface until the entire area is wet. Then brush lightly over cement surface to spread cement to a uniform coating over the entire surface. This technique is useful for large areas and/or for curved surfaces which are difficult to otherwise coat. Clean brush and tools off in warm water. If the cement has been thinned for this procedure, mark "thinned cement" on the bottle, and use for further precoat work only.

### **D. Apply Gage**

Using a sharp red pencil or metal scribe, mark the gage center-line location off to the side of the precoat. (Do not mark in bond area of gage.) Using tweezers, remove glass slide containing gage from package. Note: One end of the plastic protective package is open for gage removal. Remove tape dot over leads with tweezers while holding leads in place with thumb or finger. Place glass slide flat on table, and with tweezers or razor blade, lift the tape carrier gently and slowly off the slide until it is loose up to the window cut-outs.

Do this on both ends of the temporary tape carrier. Grasp one end of the carrier with the tweezers and lift upward until the gage is off the slide. Be sure not to apply any twisting forces into the tape as this might distort the grid, just lift upward gently until gage tape is free of the slide.

With the gage location sighted under the microscope at 7x power, hold the gage with tweezers over the surface and align gage with alignment marks previously scribed on the precoat. Be sure to position gage exactly as desired before lowering gage on to the surface. Do not move the gage around once it touches the cement surface because silicone tape leaves residue which is detrimental to good bonding. A second tweezers held with the free hand may be used to press gage down at the exact location. The tweezers holding gage tape may be released from the tape by rolling the tweezers free. Another method to press the gage in place is to press down with the finger of the second hand (dressed with surgical glove) and remove the tweezers from the tape. Using a blunt tweezers, press the tape carrier into intimate contact with the precoat.

Route the leads as required by bending them in a convoluted or "S" pattern using the tweezers to perform bend. Lay a flat foil or razor over the gage tape and press downward to hold the lead next to the grid in place and grasp the lead with the tweezers. Hold the tweezers perpendicular to the precoat and bend as required. If the tweezers are not held perpendicular to the precoat surface, the bends will cause the wire to come out of plane with the surface. Using 0.050 inch wide tape bars, placed every 0.050 to 0.1 inch, tape down the leads. Check under microscope to be sure leads and tapes are in intimate contact with the precoat. When pressing down on the tape bars with tweezers, look for any movement of the wires. There should be no movement.

If gage is a dual element gage, 0.030 inch wide tape bars should be placed over crossover strand every 0.050 inch to 0.060 inch to keep this strand in contact with precoat. Check to be sure gage strands next to leads are in contact with precoat. Because leads are thicker than gage grid wire, the strands next to the lead are often times not in contact. See Figure A-1.

#### **E. Apply Cement**

Once it is certain the gage is fully in contact with precoat, cement may be applied. Open bottle and carefully mix with spatula until uniform consistency is achieved. With a gauze pad in one hand (appropriately surgically gloved) dip 10/0 brush into cement and wipe dry in gauze pad several times until good brush wetting is achieved. Under stereo microscope of 7x to 15x take a brush about one-third full of cement and apply to gage at lead-grid weld juncture. Move from the bottle to the gage quickly because working time is very short. If one dawdles, the cement will partially dry on the brush and will not spread. When this happens, remove the cement with the gauze pad and try again. The trick is to get the right amount of cement on the brush to cover the area desired.

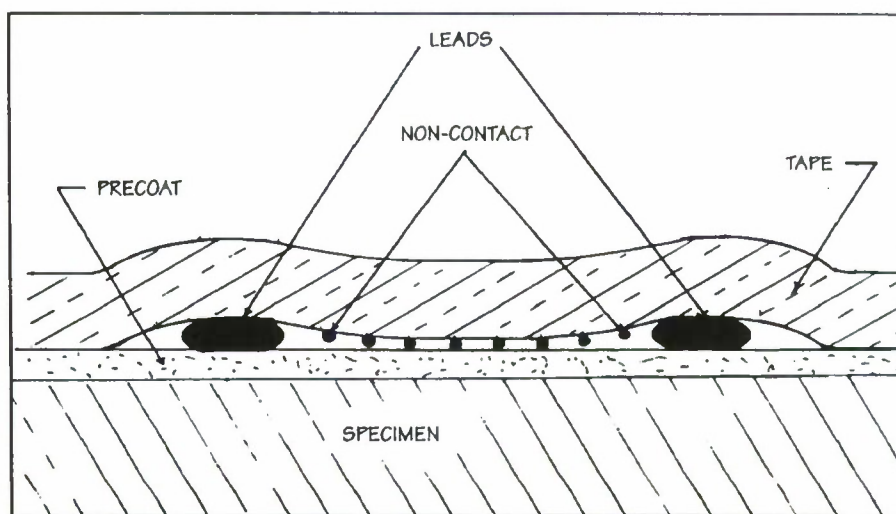


FIGURE A-1. Because of the elasticity of the fiberglass, or due to poor bonding, grid wires close to the lead wires may not be in contact with precoat.



Covering the leads takes a full brush, while covering the grid between tape bars takes a partial brush load. This technique quickly develops with practice. The grid-lead weld junction is a good starting place because, if there is too much cement on the brush, the excess may be spread over the leads. If there is not enough cement, one gets another brush load. Once the weld junction and the leads are covered, the end loops are covered next. On narrow gages, about one-third brush load usually is enough to cover the end loops. If there is too much cement on the brush, work the cement out in front of the gage away from the end loops. Apply just enough cement thickness to cover the end loops but do not touch the tape. The cement should be made to flow nicely and uniformly over the end loops and within about 0.005 inch of the tape. The cement should be thick enough so the liquid component does not migrate under the gage because it will bond to the tape adhesive. Liquid migrating under the tape can cause the tape adhesive to debond and the gage wires to lift off the precoat.

The last step is the hardest, applying cement to the grid between tape bars without cement or liquid component touching the tape or tape adhesive. Load enough cement on the brush to cover four to six strands with one pass. Too much cement on the brush will flow into the tape carrier adhesive, while too little cement will not be fluid enough to flow. The top of the grid and lead wires should just be barely covered. Do not build up any thickness over the grid wires. Yellow Cerro cement is semitransparent, and the grid and leads are visible, even though they are covered.

#### **F. Cure Cement RT = 1/2 Hour 200°F 5-10 Minutes at 350°F**

Air dry cement for 1/2 to 8 hours; longer rather than shorter air drying time is preferable. If the humidity is in the 40% range, air drying time should be at least 4 - 6 hours. Then cure the gage at 200 - 225°F for ½ to 1 hour followed by a short cure, 5 to 10 minutes at 350°F. A forced convection oven is best for this work. However for small parts, a hot plate with good temperature control is adequate. It is important to keep the parts below 400°F because at 400°F the silicone adhesive on the tape carrier begins to bond permanently to the precoat. After a 400°F cure (or higher) the tape carrier adhesive does not completely come off the precoat when the tape is removed, but instead leaves a film of adhesive particles which are difficult to remove.

#### **G. Remove Temporary Tape Carrier**

After the short cure at 350°F, remove from oven and cool to room temperature, then promptly remove the temporary tape carrier. This is done by grasping the corner of the tape bar with tweezers, peeling it back over itself and at 90 degrees to the original tape bar position. Keep the removed portion of the tape bar in the plane of the gage. Do not

lift up on the tape bar. The tape bar should be rolled off the wires with no lifting motion, so the wires are not stretched. Any lifting of the wires places very large stresses on the cement-wire bond and since the cement is not fully cured, fracture of this bond can occur.

#### **H. Bake at 800°F**

After removal of all tape, the gage should be oven baked for 2 hours at 800 to 850°F. This bake cures the cement and bakes out any silicone residue remaining from the tape carrier adhesive. Baking at any temperature lower than 800°F will not work. Caution: Be sure all teflon tape is removed before heating. Teflon emits toxic fumes that are deadly when overheated. The heat converts silicone residue to silica, which mixes readily with the cement and is not detrimental to the coating.

If HFK gages are being bonded, cure temperature cannot exceed 650°F or the self temperature compensation characteristics will be destroyed. There may also be other reasons the 800°F bake cannot be used. In this case, the carrier tape residue must be removed with solvent cleaning. This should be done immediately after tape removal. Using an artist's brush and a safety bottle with methyl ethyl ketone (MEK) solvent, brush the solvent parallel to the grid from the leads to the top of the gage. Brush in one direction only. Observe under the microscope as the brushing proceeds. Larger particles of silicone adhesive will swell from the MEK and will appear as clear jelly. These particles usually are more prevalent at the tape-ceramic interface. Often it takes a great deal of brushing to remove these particles. About 25 brush strokes are usually necessary to clean the grid area. The MEK does not dissolve the silicone, but the brushing action mechanically removes the silicone out of the grid area. If larger particles are observed, they may be removed using a sharp tweezers or pin. If, during the bonding process, the ceramic cement liquid has touched the tape adhesive it will bond to the silicone and color the silicone so it looks like ceramic cement. Observation under the microscope while brushing with MEK will reveal this colored silicone as it swells from the MEK. Once identified then it can be removed.

#### **I. Apply Final Coating**

After cleaning allow sufficient time for the MEK to fully evaporate. Test for cleanliness by checking if the cement wets the precoat. Do this in a remote area where a tape bar had been bonded. The cement should wet immediately. If it does not wet immediately, the gage area is not clean. Continued brushing with wet cement will passivate any remaining silicone and forced wetting will occur. Do not force wet because this will entrap silicone contaminants in the cement. This will also cause poor bonds and eventual voids to be created when the silicone contaminant turns to silica powder at high temperature.



If the wetting test indicates immediate wetting, the gage area can be coated with the final coat of the cement. Using the 10/0 sable brush about one-third full of freshly stirred cement quickly coat the gage grid area up to the level of the gage wire. Do not cover the grid with the first application. Wetting the grid wires is good but do not cover them. If excessive cement is applied on the first stroke, it will cause air bubbles to form under the grid and these bubbles can later turn into voids. The precoat is porous and contains many air spaces. These spaces must be filled slowly by applying thin layers of cement which will displace the air. Covering the precoat only up to the grid wires will minimize bubble formation.

Next cover the lead areas with the 10/0 brush fully loaded with cement. Be sure to work the cement under and over the leads. By this time the cement in the grid area has partially dried and settled. The entire grid should now be covered with a full brush load of cement. Work the wet cement quickly to a uniform coating over the entire gage as well as over the first coat. Apply enough cement at this time to thinly cover the grid when the cement dries. The total cement layer should be as thin as possible. Total installation thickness should be in the 6- to 10-mil range. The thinner the cement, the better the installation. Thick cement will reduce strain range and thermal shock characteristics of the installation. Thick installations will delaminate or spall off at elevated temperatures.

#### **J. Final Cure**

Air dry and observe coverage of cement under microscope. If grids are exposed, they should be covered now. The wet cement blends very well with a partially air dried layer. Air dry for 1/2 to 6 hours, depending on the humidity. Oven cure at 200 to 225°F for 1 hour, 400°F for 1 hour and 600 to 650°F for 1 hour. Cool to room temperature and inspect under microscope. If wire looks exposed, inspect under higher power, say 20x to 30x. If grid is exposed, it needs to be covered. At this stage of the process, patching is quite difficult. As cement is applied, the cured cement quickly absorbs the liquid component and the liquid starved fresh cement wants to ball up rather than spread. It is best to do any patching when the cement is fully saturated with liquid prior to the cure process. There is no easy way to patch a cured installation, except by making the installation a lot thicker. Any patches, of course, need to go through the complete cure process.

#### **VI. WATERPROOFING**

Application of waterproofing materials is not recommended. If moisture-proofing must be done, or if it is necessary to protect the installation from contaminants, then cover the installation with fiberglass teflon tape. This tape has withstood water immersion for 1 week without passing any moisture. The tape should be removed prior to heating up because teflon emits toxic fumes if heated above 500°F. In no case should moisture-



proofing materials which wick into the cement be used. If they are used, the slightest amount of moisture which is always absorbed turns to steam upon heating, generating pressures which will blow off or delaminate the gage installation. An unprotected installation will absorb moisture which will cause temporary leakage to ground on the gages. This leakage (and moisture) will quickly disappear upon heating the structure.

## **VII. QUALITY CONTROL**

Check gage resistance at room temperature and record on gage installation data sheet. Measure gage resistance to ground and record. Identify gage type, lot number, manufacturing date, gage factor, lead resistance and location where gage resistance was measured. Record cement type, lot number, and manufacturing date. Note if thinner was added and how many drops were added. Record grit blasting medium and grit size if used, type of nickel aluminide, if used. Record cleaning bake procedure, oven type, and all solvents used to clean specimen. Attach strain gage and cement data sheets to installation record log and place in job file.

## APPENDIX B

### FLAME SPRAY ATTACHMENT PROCEDURE

#### I. GENERAL

#### II. FACILITY REQUIREMENTS

#### III. EQUIPMENT

- A. Flame Spray Booth, Figure B-2
- B. Flame Spray Equipment
- C. Gasses
- D. Air Compressor
- E. Air Conditioner/ Dehumidifier
- F. Air Cooler
- G. Bench w/ Microscope
- H. Tools
  - I. Materials
- J. Grit Blast Equipment

#### IV. APPLICATION PROCEDURES

##### 1. Surface Preparation

- a. General Cleaning
- b. Heat Cleaning
- c. Grit Blasting
- d. Nickel Aluminide Surface Preparation
  - 1. Nickel Aluminide
  - 2. Equipment
  - 3. CM-1000 Flame Spray System
  - 4. Bonding Procedure using CM-1000 System

## **I. GENERAL**

The procedures described herein are intended for installation of strain gages and temperature sensors for measurements in extreme environments, in particular high temperature, high thermal shock, high "G" loads, high acoustic or mechanical vibration, impact, high erosion, high vacuum, nuclear radiation, monotonic oxygen and various combinations of these environments.

The procedure for attachment of sensors by flame spray techniques dates back to the 1950's. The procedure simply consists of:

- A) applying a thin insulating coating to the surface of the structure by flame spraying a ceramic insulating material,
- B) taping a sensor to the surface using a temporary tape carrier which has cut-outs exposing about 50% of the sensor while the other 50% is taped tightly to the structure, (See Figure B-1.)
- C) the exposed portion of the sensor is flame sprayed and attached to the precoat,
- D) the temporary tape carrier is then removed and
- E) the remainder of the sensor is embedded in flame sprayed ceramic.

The flame spraying process consists of melting a ceramic powder or solid rod in a flame, and using a jet of air, blasting the molten, atomized ceramic at high velocity against the surface. The procedure produces an extremely durable and strong coating with the sensor embedded within the ceramic matrix. The temporary tape frames used in the process are special; they must withstand the hostile environment of the flame spray shower of hot, high velocity molten particles, and must hold the fragile sensor in perfect configuration during storage, handling and application.

## **II. FACILITY REQUIREMENTS**

Because of the acoustic noise associated with the Rokide\* flame spraying process, the spraying should be done in a special room. The room should be constructed of fireproof materials in the walls, ceiling and floor, and should contain fireproof furniture. No flammables should be stored in the room, i.e.: waste baskets, trash barrels, cardboard or wooden boxes, loose paper, flammable chemicals, solvents, etc.

The room should contain good lighting, a flame spray booth with exhaust fan, a fire extinguisher, the flame spray equipment, a grit blast chamber with gun and exhaust system, a supply of oxygen and acetylene gases, air conditioner, dehumidifier and



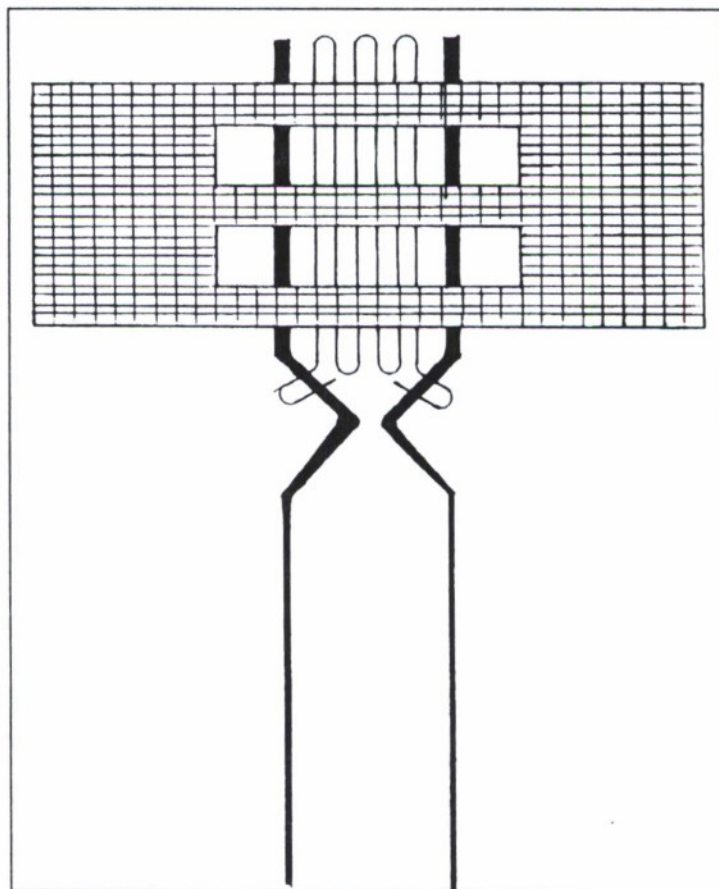


FIGURE B-1. Free Filament Strain Gage for Flame Spray Application

humidity indicator, a filtered compressed air supply, vortex air cooler, a work bench with 7x to 30x wide field stereo zoom microscope and suitable stand, and a tool box with tools and materials as outlined below.

### **III. EQUIPMENT**

#### **A. Flame Spray Booth, Figure B-2**

The flame spray booth must contain the flame sprayed particles and should be at least 4 feet wide x 5 feet high and 4 feet deep. The booth should be large enough to accommodate the largest parts to be worked on. The booth must have an exhaust system:

- A. To safely exhaust unburned combustible gasses when setting up the system, and
- B. To exhaust products of combustion, which includes ozone generated during the spray process
- C. To remove heat generated by the combustion process
- D. To exhaust the flame sprayed particles.

The exhaust system must be powerful. A 1/2 hp fan is suitable for an 18 inches diameter short duct. An automatic damper in the duct prevents back flow of cold outside air into the room. Some local codes require a water wash system to remove particulate from the air. State and local codes differ considerably throughout the country, and the facilities engineer must be guided by these and local suppliers for the equipment most suitable for the location. With a powerful exhaust system, there must be an air inlet into the room. This should preferably come from an air conditioned space, to reduce the load on the room air conditioner/dehumidifier. The booth light switch and the booth exhaust fan switch should be mounted conveniently on or near the booth.

#### **B. Flame Spray Equipment**

The flame spray equipment should consist of a Rokide ceramic spray system (Figure B-3) and a powder metal spraying system (Figure B-4) as a minimum. The metal spraying powder system is used to apply nickel aluminide precoat as a surface preparation. It is a standard procedure specified by many Rokide users. This program used the Hitec CM-1000 powder system consisting of a mini-gun, (Figure B-5) a powder feeder (Figure B-6) and a control panel. The system requires a small acetylene tank, a standard oxygen tank and an air supply. A 2 hp 20-gallon tank air compressor is adequate to operate this system. An oilless compressor is preferable, because it eliminates the need for expensive oil filters. The Rokide system consists of a spray unit, either Norton or Miller, a compatible flow meter, oxygen and acetylene regulators, and a filtered air supply. Although filtered shop air is, in many places, adequate, it is more often not adequate. Inadequate air line pressure, or pressure fluctuations are a principle source of equipment malfunction. Any sudden air line fluctuation may cause rod "spitting" which might damage a strain gage at a critical point in the process.

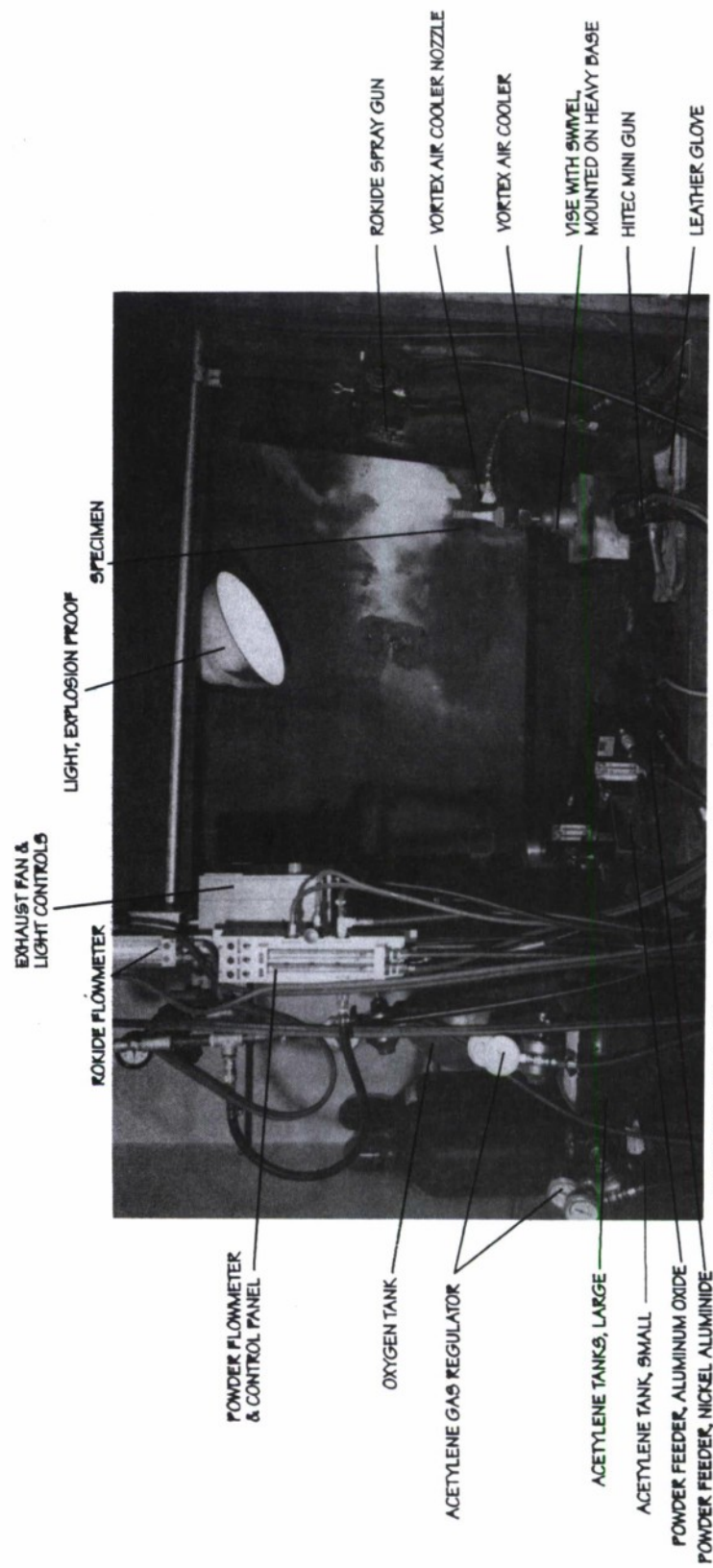


FIGURE B-2. Flame Spray Equipment Mounted In Spray Booth



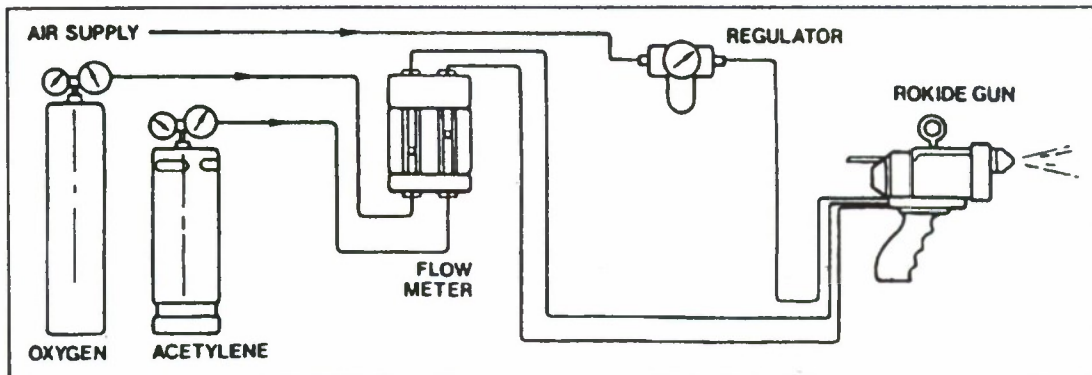


FIGURE B-3. Schematic of Rokide Flame Spray Equipment

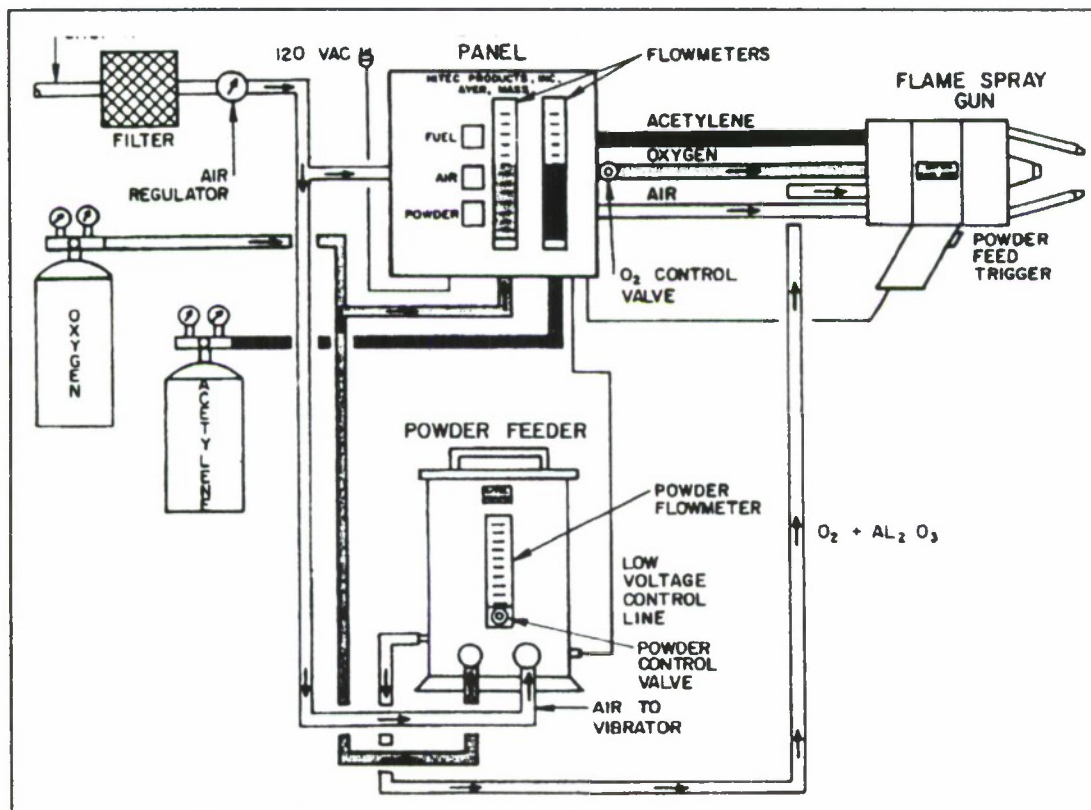


FIGURE B-4. Schematic of CM-1000 Powder Flame Spray Equipment

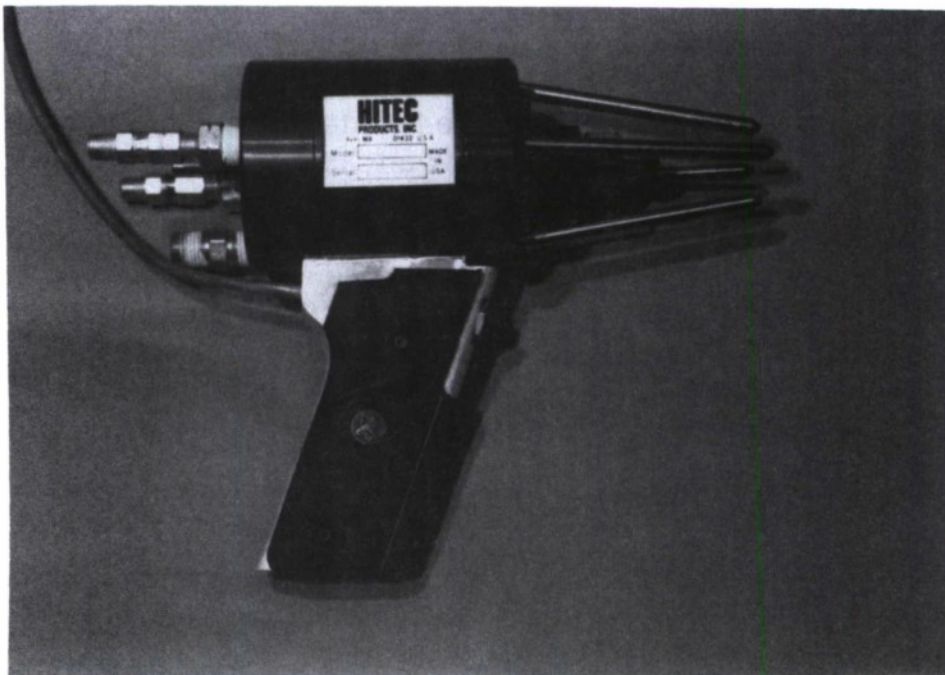


FIGURE B-5. Hitec Mini-Gun Powder Flame Spray Unit

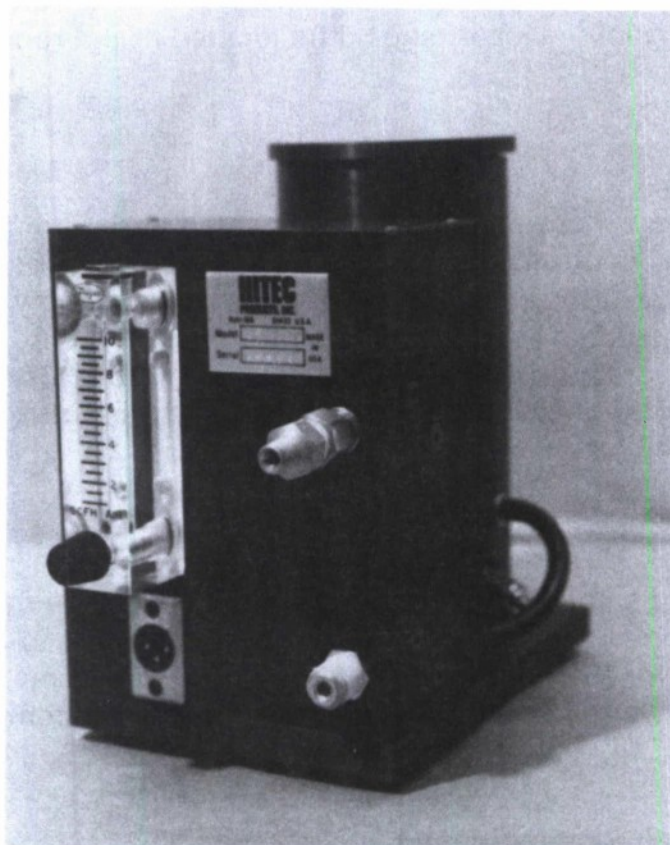


FIGURE B-6. Powder Feeder for CM-1000 Flame Spray System

### **C. Gasses**

Oxygen and acetylene gasses can be located in the Rokide room or just outside the room. One large acetylene tank and one large oxygen tank are adequate for occasional spraying. For continuous work two or three tanks should be manifolded together to eliminate the time loss of frequent tank changes. The tanks may be placed outside the room but not in the cold. The tanks should be at least 68°F, otherwise liquid acetone will be drawn off from the acetylene tanks. Shut gun off immediately if the flowmeter balls start to float in clear liquid. This may also happen when using a single tank and the tank pressure approaches 50 psi. Do not use acetylene tanks which have been brought in from the cold until the tanks warm up to room temperature.

### **D. Air Compressor**

To eliminate the expense of lost time and lost gages, a dedicated compressed air system is used by most. A 10 hp compressor with 120-gallon tank is a minimum unit that will do an adequate job. Since larger compressors are not oilless, a good filter system is necessary. Because of noise, the compressor is usually placed some distance away from people, but in a warm clean area.

### **E. Air Conditioner/ Dehumidifier**

Air conditioner/dehumidifier should be sized to maintain room humidity below 45% R.H. at all times. A relative humidity indicator should be located in the room and frequently monitored.

### **F. Air Cooler**

A vise or specimen holder is an essential tool to be located in the booth. A vortex type air cooler with variable flow control valve and pressure gage is essential for good installations. It also improves efficiency and saves rods by reducing waiting time for the specimen to cool. A magnetic based holder is used to clamp the vortex cooler to the side of the flame spray booth near the specimen vise. (See Figure B-2) The vortex cooler must be operated on ultrafiltered clean air. If an oilless compressor is available, it should be used to supply air to the vortex cooler.

### **G. Bench w/ Microscope and Tools and Materials**

A work bench with a clean, smooth laminated top, a tool box and a microscope are essential for this work. The microscope should be a wide field, 7x to 30x stereo zoom type with a stand suitable for the type of work to be done. Very small specimens require only the simplest of stands, while large pieces will require a more complex, adjustable stand.



## H. Tools

The tool box should contain the following tools:

1. Small vises (2)
2. Hargrave welders clamps, large (2), small (2), very small (2)
3. Hand held vises (2)
4. Screw clamps-assorted sizes from 1 inch to 6 inches
5. Dental pick - straight
6. Pin pick - homemade from artist's brush and sewing needle
7. Metal scribe
8. Carbide tip scribe in aluminum holder
9. Tweezers, Dumont 3 C (2)
10. Tweezers, Rio Grande style 5
11. Tweezers, self clamping
12. Scissors, 4 inches embroidery, Wiss
13. MEK safety dispenser, 3 oz
14. Tape dispenser, transparent
15. Tape dispenser, masking
16. Tape dispenser, mylar
17. "Q" tip holder
18. Tool stand, rotary
19. Artist's brush, sable, 10/0
20. Industrial razors, package, single edge
21. Dresser stick, aluminum oxide, white, 1/4 inch x 1/4 inch x 4 inches, 60 grit
22. Pencil, red
23. Pencil, silver
24. Scale, machinist's, 6 inches,
25. Gauze pad holder, 2 inches x 2 inches
26. Microscope light
27. Eye protection, clear with side shields
28. Eye protection, no. 5 shade with side shields
29. Ear protection
30. Igniter
31. Rubber mallet
32. Welder's gloves, leather
33. Shop coat - cotton
34. Adjustable wrench, 10 inches (1), 8 inches (1), 6 inches (1)
35. Open end wrenches, 11/16 inch, 5/8 inch, 9/16 inch, 1/2 inch, 7/16 inch
36. Pliers, general purpose
37. Vise grips, assorted sizes (3), sheet metal (1)
38. Glass plate, 2 inches x 6 inches double thickness pane (3)
39. Spatula, stainless steel, 6 inches
40. Multimeter, Beckman 850 or equivalent

## **I. Materials**

1. Rod, Hitec HT, high purity alumina
2. Rod, Hitec S, standard alumina
3. Powder, nickel aluminide
4. Tape, transparent
5. Tape, masking
6. Tape, aluminum/teflon, 1/2 inch wide, No. 404
7. Tape, fiberglass teflon, silicone adhesive, 1 1/2 inches wide, No. 64 or equivalent
8. Tape, silicone, SA, 1 inch wide
9. "Q" tips, wooden
10. Gauze pads, 2 inches x 2 inches, sterile
11. M.E.K.
12. Alcohol, Isopropyl
13. Strain gages, assorted
14. Thermocouples, convoluted, C/A, 0.003 inch dia. and 0.005 inch dia.
15. Paint, red, model makers
16. Cement, Ceramic, Yellow Cerro, 1-oz bottle

## **J. Grit Blast Equipment**

The grit blast equipment can also be located some distance away from the Rokide room. This equipment should consist of a blast cabinet large enough to accommodate the largest parts to be grit blasted. The unit should have a pressure blast generator, good lighting, and a suitable exhaust system. Contrary to common practice, grit should be used once only and should not be recycled. The blast generator must be located near a supply of clean compressed air.

## **VIII. Application Procedures**

### **I. Surface Preparation**

#### **a. General Cleaning of Specimen**

Remove all surface contaminants, grease, oil, paint, protective coatings, rust, scale or other contaminants with solvents or cleaners. Rust scale and burrs may be removed using wire brush, grinder/sander, file or stone.

#### **b. Heat Cleaning**

Once obvious surface contaminants are removed, the part should be baked for 2 hours at 800°F to burn off contaminants absorbed into pores, crevices, cracks or rolled into the material during the rolling process. Some titanium alloys and super alloys that look clean are not.

#### **c. Grit Blasting**

In order for flame sprayed alumina coatings to adhere, the surface must be roughened and cleaned using a sharp aluminum oxide or silicon carbide grit blasting. The grit size used depends on the time at temperature the part will experience. See graph, Figure B-7.

For large parts made from super alloy material that will be exposed to 800°C or higher, a no. 30 or no. 46 aluminum oxide grit used in a pressure blast generator will do a good job. The grit blast will raise the surface about 2 mils. The grit blasting process greatly increases surface area for bonding. To minimize residual stresses, blasting at about a 30-degree angle from the horizontal will introduce minimal surface stresses. Direct blasting at 90 degrees to the surface will generate compressive stresses, which will cause thin metals to curve downward. Grit striking at various angles will generate stress gradients causing thin parts to warp, pucker or bend in various directions. For this reason, grit blasting should only be used on robust parts where residual stresses are of no consequence. Compressive residual stresses are sometimes desirable for applications where repeated cycling stresses will be applied. In this case, grit blasting at 90 degrees to the surface is appropriate.

If the part will not experience very high temperatures, and coarse grit blasting is not desirable, smaller size grit will work quite well. Grit blasting with an S.S. White Airbrasive machine with 50-micron aluminum oxide powder has produced a good surface for flame sprayed coatings used at moderate temperatures.

Roughening the surface by hand, using coarse abrasive paper or cloth does not produce a good surface for flame spraying. Machine sanding or grinding does not produce a suitable surface either, but these procedures do produce severe residual stresses.



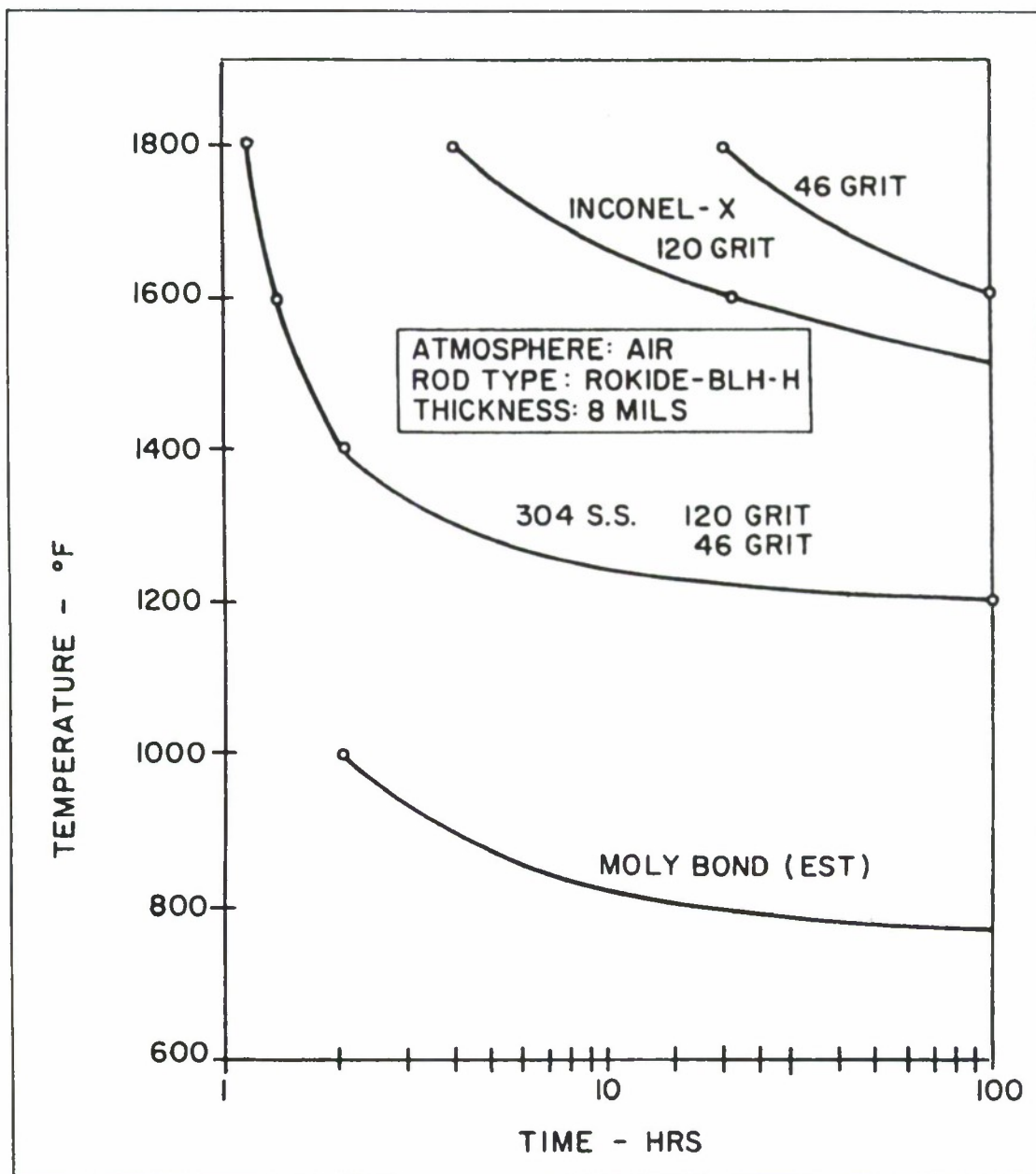


FIGURE B-7. Strain Gage Installation Life At Elevated Temperature

#### **d. Nickel Aluminide Surface Preparation**

When grit blasting is not allowed, a surface texture similar to coarse grit blast can be achieved by flame spraying nickel aluminide. Nickel aluminide applied 2-3 mils thick produces an excellent surface for flame sprayed ceramic coatings while introducing essentially no residual stresses. This method has become standard for many applications where thin-walled parts, such as small air cooled turbine blades must be instrumented but where no distortions are allowed. Even thin shim stock can be coated with a minimum of distortion.

##### **1. Nickel Aluminide**

Nickel aluminide flame spray powders consist of fine, nickel coated aluminum particles, sometimes with chromium additions that bond extremely well to most metals. The particle, once heated begins a synergistic, exothermic reaction between the nickel and aluminum which generates additional heat while in transit to the specimen surface. The particle produces an extremely tenacious bond which is alloyed with the specimen surface. The nickel aluminide with chromium additions produces hard, oxidation resistant coatings usable to about 1000°C. These coatings do not produce the local annealing which occurs from spot welding, with subsequent reduction in fatigue life. The bond is metallurgical, the base metal being affected to a depth of about one microinch<sup>67</sup>. Nickel aluminide coatings are used as a base coat for metal sprayed nichrome (80 Ni 20 Cr) to which spot welds can be made without damage to the base metal. The nickel aluminide is also used as a surface preparation for flame sprayed ceramic such as aluminum oxide. It can be applied to very hard materials that are not easily grit blasted, such as thin, air cooled turbine blades or thin sheets of foil without distortion.

##### **2. Equipment**

Various metal spray systems are available which produce excellent coatings of nickel aluminide. Each system has its own operating instructions. The surface coatings produced on this project were done using a Hitec CM-1000 powder flame spray system and the procedure given here applies to that system.

##### **3. CM-1000 Flame Spray System**

The model CM-1000 is a miniaturized powder flame spray system developed for sensor application. The system consists of a mini-gun, a powder feeder, and a control panel. The system uses an oxy/acetylene flame to melt ceramic particles and compressed air to propel these particles to the surface. The gun is pressure fed, not gravity, and will spray with the gun pointed in any direction, including vertical. Oxygen is used as the carrier gas and is consumed during combustion, eliminating the laminar gas film coating the particles and therefore improving heat transfer into the spray particles. Because of miniature size and fine control the system is especially well suited for spraying nickel aluminide precoat, where accurate control of thickness is essential.

#### 4. Bonding Procedure Using CM-1000 System

Set the system up as follows (refer to operation manual for full explanation of operational and maintenance procedure):

Oxygen regulator pressure:	20 psi
Acetylene regulator pressure:	6-10 psi (Start at 7.5 psi)
Air pressure:	45 psi
Oxygen flow:	2.5 points (center of top ball)
Acetylene flow:	6.0 points (center of top ball)
Powder flow:	5.0 points

Nickel Aluminide powder must be kept clean and dry. If powder is accidentally spilled it should be discarded. Prior to loading powder into hopper, alternately roll can on a flat surface and slowly turn it end over end several times before loading the hopper. Do not load more powder than you expect to spray at one time. If, at any time, the powder does not flow easily from the can into the hopper, pass the powder through a 200 mesh screen while loading hopper. Always replace cap tightly on powder can and store in dry cabinet. Do not attempt to save or store powder in hopper, nor put powder back into can after spraying. Dispose of excess material according to local regulations.

Clean specimen surface as for grit blasting. The surface need not be grit blasted, just clean. Nickel aluminide will bond to smooth or polished surfaces, as long as they are clean. Mask off the bonding area using teflon fiberglass or teflon aluminum tape. Do not touch the bond area with tape or with fingers at any time. Protect the rest of the specimen with suitable shielding, such as sheet metal foil or tape to prevent inadvertent coating with nickel aluminide, which is very hard to remove. The practice of taping the whole surface and cutting a bond path into the tape, then removing the cutout tape path is poor practice because it leaves a contaminant film of silicone adhesive which is detrimental to good bonding. A good curved path can be achieved using the teflon aluminum tape which is easily curved. On thin sheet metal parts apply cooling air to bond surface prior to application of coating.

Using the spreader nozzle directed at 20 degrees from horizontal, turn vortex air cooler to 30 psi. Turn on exhaust fan, put on eye protection with no. 5 shade lens, ignite flame and final adjust all settings. Test spray pattern on a sample piece of metal. Apply coating to specimen by spraying at a 12 inches distance to specimen. Hold gun perpendicular to surface and make quick (1/3 second) passes with a dwell off the specimen of at least 5 seconds between passes. Make three or four passes, then using fingers carefully, feel the specimen temperature (not on bond surface). The specimen should never get hot. If heating occurs, increase dwell time between passes. On large structures the heat of the gun must be directed away from the specimen, the equipment, tanks, and hoses.



Monitor coating thickness buildup by observing percentage of specimen coated. At first, just a few specks appear on the surface, followed by increasing density until nearly complete coverage occurs. This may be difficult to observe, depending on lighting, and light from the flame, moved in front of the specimen, is the best way to observe coverage. Stop the coating process at about 95-98% coverage. A very little bit of shiny specimen surface should be observed through the coating. Do not continue spraying until 100% coverage is achieved, because as thickness builds up, residual stresses increase also. Try to achieve one layer of particles bonded to the surface. If more than one layer is deposited, the particles on the second or subsequent layers fuse to each other and upon cooling shrinkage causes deformation of the part. This becomes very evident on thin materials such as foil, which will curl upon cooling if thickness exceeds one layer of particles.

When 95% to 98% coverage is achieved, shut off equipment and air cooler. Inspect coating under microscope. Using carbide tipped scribe, pick at the coating to be sure bond is strong. Remove tape from part, and with an industrial single edge razor blade, scrape coating to remove any loose particles and buildup around the tape edges. Hold razor at 90° to the surface. Blow off any loosened particles using clean air (Wear eye protection). The part is now ready for masking for cement or flame spray precoat application. If parts need to be stored, place them in a clean cabinet or cover precoat with gauze pad and place specimen in sealable plastic bag.

## **ROKIDE EQUIPMENT OPERATION**

### **General Equipment Operation**

The equipment used during this program consisted of a Norton Rokide spray unit complete with flowmeter, oxygen and acetylene regulators, and a vortex air cooler mounted in a spray both as shown in Figure B-2.

The two important aspects of obtaining good Rokide installations are:

1. thorough familiarity with the equipment, its idiosyncracies; keeping the equipment maintained and operational; and staying current with frequent practice, and
2. using the equipment for the installation of strain gages.

While veteran flame spray equipment operators are thoroughly familiar with the equipment, its operation and maintenance, the use of the equipment for sensor installations is, on a number of critical fundamental commandments, operated directly opposite to what a coatings applicator has been taught. We have found, from operating sensor installation classes, that veteran flame spray operators have their operational procedures so ingrained into their psyche that it is impossible for them to accept sensor application techniques. Two examples 1) coatings people spray at 3-4 inches distance while sensor application is done at 10 inches and, 2) for coating work the gun is run at the highest speed possible, while for sensor applications the gun is run at the slowest speed possible. Therefore, although there are exceptions, it is easier to train a strain gage technician to operate flame spray equipment than to train a flame spray coatings technician to install strain gages. At no time should this equipment be operated by a technician who has not been properly trained in the use of the equipment.

### **Technician Training and Skills Maintenance**

The procedures developed during this program have been greatly improved and simplified over procedures of the past. Employment of these procedures should result in better installations by technicians at all levels of competency. Using the procedures developed during this program will result in a significant improvement in quality, durability, and reliability, while reducing the degree of difficulty for the technician.

Technicians skilled in the art should be able to adopt these procedures from reading the text. Newly trained technicians are being taught these procedures in our training classes. Technicians once taught must stay current. To stay current a technician should operate the equipment at least three times a month. If an extensive layoff is unavoidable, then a check-out with an instructor is necessary for safe operation of the equipment.

## Equipment Description and Operation

The Norton Rokide spray unit uses an oxy/acetylene flame to melt solid 1/4 inch diameter ceramic rod. The acetylene regulator is set at 15 psi and oxygen at 90 psi. An air turbine drives a set of feed rolls which grasp the rod and move it into the flame. The rate of rod feed is adjusted by controlling air turbine speed. An on-off set of buttons turns the gasses and air turbine on. The bottom button has two positions. The first click turns on a pilot light flow of acetylene which is ignited with an igniter. This flow may be seen as a very small movement of the acetylene flowmeter ball. Once ignited, further uniform pressure on the start button increases oxygen and acetylene gas flow in correct proportion until full flow is achieved. At this point the button clicks to the "on" position and stays on until the "off" button is pressed. The "on" button also turns air on. Some of the air runs the air turbine which drives the rod into the flame. Air is also used to strip the molten ceramic particles from the rod and propel these particles at sonic velocity, toward the specimen to be coated. The flowmeter is used to monitor gas flow. An air pressure regulator and pressure gage is used to adjust and monitor air line pressure. The gas regulators are used to adjust and monitor gas pressures. The gun is turned off by pressing the "off" button. This automatically turns off all gasses and air. The rod drive engage lever is released and the hot tip of the rod is pushed out of the nozzle by pushing on the cool end of the rod. A hot ceramic tip, if left in the gun, would erode the nozzle inner diameter. When the tip cools, break off about 1 inch of molten tip using pliers or other suitable tool.

### Preignition Check List

Prior to starting the gun, the system should be checked for gas leaks at all connections using a soap and water solution or other suitable leak check. Sniff the gun nozzle to detect any smell of acetylene which may pass by the "on/off" valve "O" rings. If acetylene smell is detected, perform maintenance as required in gun maintenance manual. If no leaks are detected, turn off gasses and adjust gun for sensor operation:

1. Adjust feed roll pressure to 7 lb force on the rod. (See gun operations manual for procedure.)
2. Adjust gun feed speed. Be sure gases are off when performing this procedure. For Norton gun, with 10 feet of air hose between gun and air pressure gage, set air regulator to 90 psi when gun is full "on". Turn gun off. Position rod at tip of nozzle, lock in place, and using a stopwatch, turn gun on for exactly 60 seconds. Measure length of rod movement in 60 seconds using a 6 inch rule. Adjust air turbine speed until a rod speed of 3 1/2 inches per minute is achieved. Check this speed to be sure it repeats.

Next, position rod so the tip of the rod is flush with the end of the nozzle. (The nozzle is on the inside of the air cap. Do NOT position rod flush with end of air cap!) This is easily accomplished by extending rod out of the air cap slightly and pushing it back into gun using the end of another piece of rod until it touches the nozzle. Then lock feed



rolls. Before starting system each day remove air cap and inspect for cleanliness. Clean if necessary. (See maintenance manual.) Build-up of sprayed materials on the inside of the air cap will cause a reduced gas flow which may be observed on the flowmeters. If the flowmeters indicate reduced flow, shut off gun and clean air cap.

Before igniting gun, put on shop coat, eye protection with side shields, and ear protection; start exhaust fan, press start button for pilot flow, ignite, turn gun full "on". The gun will run momentarily without spraying until rod is advanced into flame and the spraying will begin. Check all gages for proper settings and flowmeter for uniform flow.

### **Adjust Rod Feed Speed**

Rod speed should be adjusted for each new lot of rods, because each batch of high purity rod runs at slightly different speed. Because of the narrow speed range of high purity rod, this adjustment is much more critical than for rod used in coating work. With the rod pre-set for 3.5 inches per minute, the rod should be spraying. Slowly reduce rod feed speed until a pulsation appears in the spray pattern. Then increase speed slowly until pulsation disappears. This is the correct speed for the rod lot in use. Turn off gun, open feed roll drive and immediately advance rod out of nozzle.

### **Preoperation Checklist**

1. Turn on oxygen tank and adjust regulator to 90 psi.
2. Turn on acetylene tank and adjust regulator to 15 psi. (Do NOT open tank valve more than 3/4 turn. In case of emergency the valve should not be open more than necessary.)
3. Adjust air pressure to 90 psi. Air should never be turned "off". (If gun is ignited with air "off", a meltdown of the gun front end will occur in seconds!)
4. Sniff for acetylene leaks at gun.
5. Adjust rod position flush with end of nozzle. Lock feed rolls.
6. Check to be sure feed rolls are locked.
7. Put on protective gear: shop coat, eye protection, ear protection, and the same for bystanders.
8. Turn on exhaust fan, press "on" button to pilot position, ignite pilot light, turn gun full "on", observe spray pattern. Adjust rod feed speed if necessary. Check all gages and flow.

### **Safety**

A properly trained operator using his check lists can operate this equipment safely and enjoyably. But the operator must be properly trained and completely knowledgeable in the operation and safety procedures and he must stay current. Like flying an airplane, improper operation of the equipment can result in a severe accident.

1. Gas leaks: Check for and repair any gas leak before operating equipment. See gun Operations and Maintenance manual for gas leaks in the spray unit. Do NOT operate equipment with gas leaks, no matter how small.
2. Rod start position: Rod must be flush with end of nozzle (not air cap). Failure to properly position rod may cause spalling or spitting of hot chunks of rod causing damage to air cap, operator or bystander.
3. Rod fit: Be sure rod passes through gun prior to use. Pass rod all the way through the gun and check for hang-ups. If rod jams, remove it. Check nozzle I.D. with 0.2510 inch dia. reamer. If reamer does not go through, ream nozzle I.D. with same reamer. Each rod should be checked by passing it through the gun. It's better to check it before hand than have rod hang up in the middle of spraying.
4. Lock feed rolls: If not locked, air pressure at nozzle will blow rod out of gun followed by flame which will burn operator and melt down back end of gun.
5. Clothing: Operator and all bystanders should wear fire resistant cotton or wool clothing. Denim is good; leather is very good. Always wear something that you don't mind if a hole is burned into it. Holes will be burned into clothing, because occasionally, malfunctions will occur.
6. Eye protection: Use clear goggles with complete side shields. When malfunctions in gun operation occur, white hot pea size particles of molten ceramic bounce around at pellet gun velocities - and these particles may come at you from any angle.
7. Flammables: Remove from room. Do not keep waste baskets with paper, paper cups, pads, note books, paper cartons, boxes, solvents or wooden boxes in room. Hot, molten ceramic particles will ignite anything that is ignitable.
8. Ear protection: Ear muffs or ear plugs must be worn by operator and bystander. Sonic velocity of spray nozzle produces noise well above recommended OSHA safe levels and ear protection is required by law.

### Trouble Shooters Checklist

Some of the more common problems associated with Rokide flame spray equipment are discussed here. For a complete trouble shooter's checklist, check equipment operator's maintenance manual.

1. Rod Spitting
  - A. Rod feed speed too fast/slow
  - B. Feed rolls too tight - causing rod breakage
  - C. Air line pressure fluctuations
  - D. Inadequate air pressure to gun - pressure too low
  - E. Plugged gas hole in nozzle
  - F. Build-up on air cap
  - G. Speed control failure

2. Change in Spray Pattern

- A. Plugged gas hole in nozzle
- B. Build-up on air cap (Note reduced flowmeter flow.)
- C. Air pressure fluctuation

3. Rod Feed Stops

- A. Particles blowing back through nozzle inner diameter pack around rod cause jamming. Measured impact with rubber mallet on end of rod usually will start rod moving again. Feed rolls may be too loose, or nozzle inner diameter may be worn. Usually the problem is undersize rod and the problem will disappear once undersize section is used up. If problem is persistent, replace nozzle liner or nozzle.



## **BONDING FREE FILAMENT GAGES USING ROKIDE**

### **Masking**

Remask perimeter of gage and lead wire path using one layer of teflon fiberglass tape or teflon aluminum tape. If the lead path needs to be curved, use teflon aluminum tape which may be easily stretched and contoured. Do not touch bonding surface with tape or fingers. Never lay the tape on its edge. Since the tape is used for masking, the factory edge is straight and handy to use. If laid on the bench the adhesive edge picks up dirt and debris which ends up in the edge of the Rokide coating. Use a tape dispenser to keep the tape edge clean. Protect all other areas of the part by shielding it with metal foil, sheet metal, or masking tape.

### **Precoat Application**

Place part in spray booth at a convenient position for direct access with the spray gun. Spraying should be done with gun held perpendicular to surface to be coated. Small parts can be held in a vise. A vise on a swivel base is convenient for positioning parts for easy access. The vise should be mounted on a heavy base, so it will not be overturned by the high velocity blast of air from the spray process. If the part is thin, apply vortex cooling on the area to be sprayed.

Put on shop coat, eye shields, and ear protection. Position rod in nozzle, lock feed rolls, check gas and air pressure, turn on exhaust fan, press "on" button to pilot position; ignite, then press to full "on" position. Spray at 8-10 inch distance. Note the dense concentration of spray particles, or cone, at center of the spray pattern. Direct this dense cone at the bonding surface making a 1 second pass over bond area, then resting for a sufficient time to allow specimen to cool. During the precoat, the specimen should not exceed 125°F. (You should be able to touch the specimen.) Always be aware of the dense cone pattern in the center of spray; the pattern is about 1/2 inch diameter. If this pattern disappears, stop spraying and check for plugged nozzle or build-up on air cap. Usually, build-up on air cap is the cause of the problem.

Apply approximately 2-3 mils thick precoat. It should look grey, not white. If precoat turns white, it is too thick. Turn off gun and examine precoat under microscope. Base metal should still be visible through the precoat. It takes a practiced eye to know exactly when enough precoat is applied. The tendency is to apply too much material. Remember, the gage is supported on the peaks of the particles. Subsequent coatings will pass through the grid and fill in the valleys under the gage wire.

## Apply Gage

Remove all excess particles of over-spray on the tape perimeter using tweezers or fingers. Using a sharp red pencil, mark the gage location center line off to the sides of the precoat. (Do not mark in bond area of gage.) Using tweezers, remove glass slide containing gage from package. Note: One end of the plastic protective package is open for gage removal. Remove tape dot over gage leads with tweezers while holding leads in place with thumb or finger. Place glass slide flat on the table and with tweezers or razor blade, lift the tape carrier gently upward until it is loose up to the window cut-outs. Do this on both ends of the temporary carrier. Grasp one end of this carrier with tweezers and lift gently upward until the gage is off the slide. Be sure not to distort the grid in any way during this lifting process.

With the gage location sighted under the microscope at 7x power, hold the gage with tweezers just over the surface and align gage with scribed alignment marks. Hold the gage in exact location before lowering it onto precoat. Do not move gage around once it touches the precoat, because silicone tape leaves a residue which is detrimental to good bonding. Also, tape which is moved from place to place quickly loses its stickiness and will later debond during flame spraying. A second tweezers held in the free hand may be used to press the gage down at the exact location. The tweezers holding the gage may be released from the carrier by rotating and pulling them free. Another method of pressing the gage in place is to press down with the finger of the free hand, (appropriately covered with surgical glove), onto the grid area pressing it down onto the precoat. While the grid is held down, the tweezers may be rotated and pulled from tape. Using the blunt tweezers, press the tape carrier into intimate contact with the precoat.

Route the leads as required by bending them into a convoluted or "S" pattern using the tweezers to perform bend. Place a piece of foil or razor over the gage tape carrier and press down on the razor with one finger to hold lead firmly in place while bending with tweezers. See Figure B-10d. Hold the lead with the tweezers perpendicular to the precoat and bend as required. If the tweezers are not held perpendicular to the precoat surface, the bends will cause the wire to come out of plane with the surface. Using 0.050 inch wide tape bars, placed every 0.050 inch to every 0.1 inch apart, tape down leads. Check under microscope to be sure leads and tapes are in intimate contact with precoat. When pressing down on the tape bars with tweezers, look for any movement of the wires. There should be no movement.

If gage is a dual element gage, 0.030 inch wide tape bars should be placed over crossover strand with 0.050 inch to 0.060 inch spacing to keep this strand in contact with precoat. See Figure B-10c. Check to be sure gage strands next to leads are in contact with precoat. Because leads are thicker than gage grid wire, the strands next to the lead are often times not in contact. See Figure B-8.

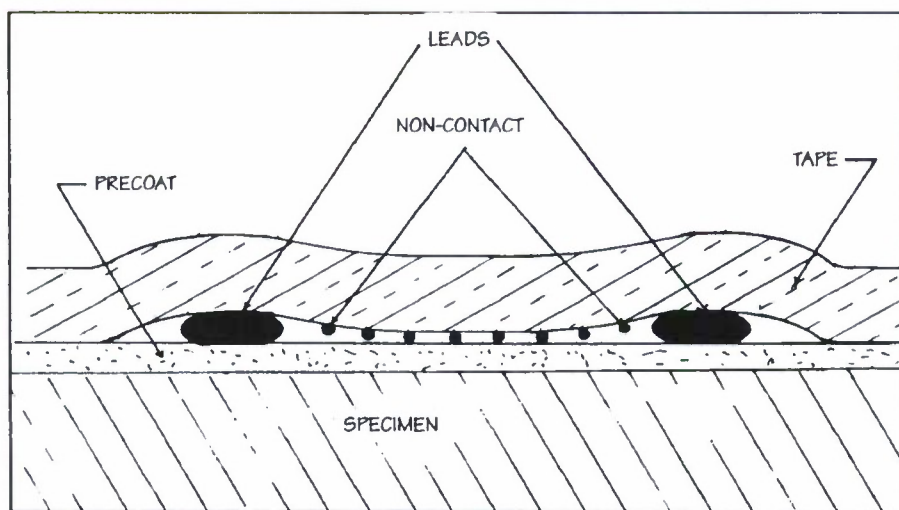


FIGURE B-8. Cross Section of Gage Showing Some Grid Wire Strands Lifted From Precoat



The next step is to place wider tape bars directly over the gage grid and lead tape bars. This second set of tape bars should over-hang the initial tape bars by about 0.005 inch on each side. See Figure B-9. Therefore, the tape width is about 10 mils wider than the initial tape bar. Since 0.050 inch wide bars were selected to hold leads in place, they should be covered with 0.060 inch wide tape bars. The gage grid usually has 0.030 inch or 0.040 inch wide tape bars. These should be covered with 0.040 inch and 0.050 inch wide tape bars respectively.

The purpose of these additional tape bars is two-fold:

- A. The additional thickness protects the adhesive from the heat of the process, and
- B. The 5-mil overhang causes the sprayed material to taper off and end without touching the tape adhesive. The taper produces a uniform blend between tack coat and cover coat. The need to break off any sharp corner at the tape bar is eliminated. Also eliminated is entrapped adhesive, which always was a problem using the old technique whereby the spray particles extended to the edge of the tape. (Some of the Rokide bonded to the adhesive, holding it in the crevices rather than letting it come off with the tape carrier.)

### **Making Tape Bars**

The tape bars can be made as follows:

Using 2 inches x 6 inches double thickness pane glass, bond about a 5 inches length of teflon-fiberglass tape. Place a second layer of tape exactly over the first layer. Total thickness of tape should be 12 to 16 mils. Using a 6 inch steel rule and a single edge industrial razor, cut a small quantity each of tape bars 0.030 inch wide, 0.040 inch wide, 0.050 inch wide, 0.060 inch wide, 0.100 inch wide. Another 6 inch rule, graduated in 1/100 inch can be used as a measure. Or, the glass slide can be mounted with small pieces of double-sided tape to the bed of an X-Y machinist slide. The rule can be mounted in a holder perpendicular to the tape and the slide traversed in exact width increments desired by turning the micrometer wheel. The razor should be held at exactly 90 degrees to the surface in order to make tape bars without under-cut. The tape bars can be cut to the length desired using scissors. The length should be equal to or slightly longer than the first set of tape bars.

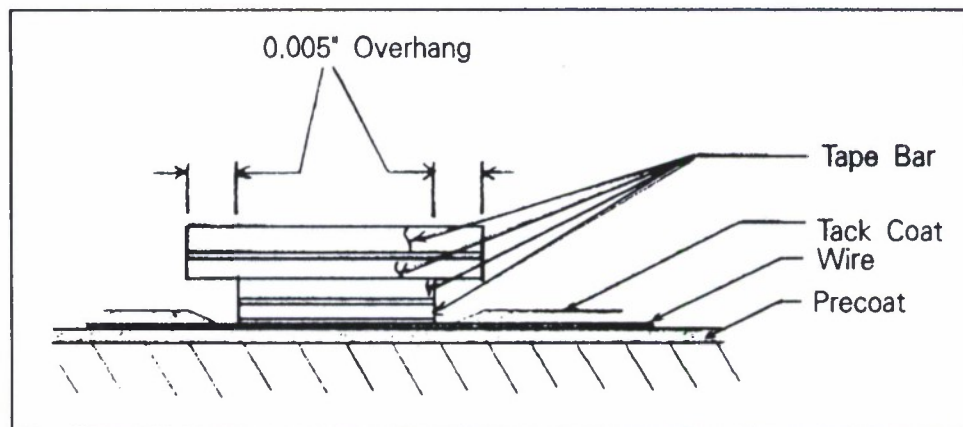


Figure B-9. Cross Section of Free Filament Gage Prepared for Flame Spray Installation

### Masking Gage Perimeter

Once all the grid and leads are double taped, place a single thickness piece of tape across end of gage 0.030 inch to 0.100 inch beyond end loops, and a second piece of tape over end of the lead area. Check gage continuity with multimeter and check resistance to ground. Apply single thickness teflon fiberglass tape on each side of gage leads, about 0.030 inch away from leads. Inspect under microscope at 7x power. Press on all tape bars to be sure grid wire and leads are in intimate contact with the precoat. If there is wire movement when tape is pressed indicating debonding from precoat, check again in a few minutes. If debonding is evident there are two methods to overcome this problem:

1. Apply spring pressure over tape using a 0.010 inch thick x 0.050 inch wide x 1 inch long steel spring made from shim stock. Tape one end of bar using teflon aluminum tape; locate over the loose tape bar and bend steel bar so it will apply vertical pressure to tape. See Figure B-10e. Tape the other end of spring to specimen just beyond the edge tape. Check for movement when pressing down from top. If wire is firmly against precoat, proceed with coating application. If it is not, continue with repair.
2. The second method of repair is to replace the defective tape carrier with a fresh tape carrier. Cut strips of teflon aluminum tape wide enough to fit between gage grid tape bars but not to exceed 0.040 inch. Place these tape bars between original tape bars (second layer of covering bars having been first removed) and press in place. One tape bar will go over gage end loops. Separate original tape bars from carrier tape by cutting with razor and removing carrier end piece (See Figures B-10A and B-10B). Remove original tape bars by grasping with tweezers and rolling off at 45 degrees to original position while holding down adjacent teflon aluminum tape bars by pressing down with blunt tweezers. Remove all original grid tape bars. The teflon aluminum tape is very strong because it does not contain parting film (as is used with the teflon fiberglass tape. The parting film makes it easy to unwind from the roll). The teflon aluminum tape uses a cloth liner instead of parting film, therefore it has much greater bond strength to precoat. The teflon aluminum tape also conforms to the vertical irregularities of leads and grid better because the annealed aluminum, once bent, will hold its bend shape, unlike fiberglass which always tries to straighten out. Cover the teflon aluminum tape bars with the second layer of tape bars (use teflon fiberglass) extending 0.005 inch beyond each side of tape width, as was done previously, and complete border taping as before. Inspect for bonding under microscope. If no debonding is evident, proceed to tack coat procedure.



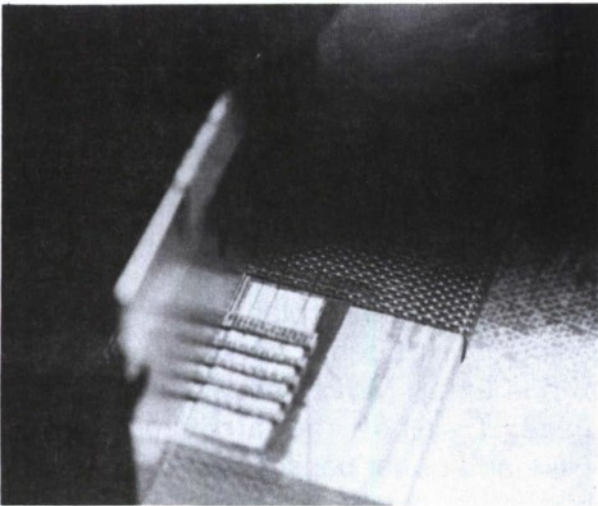


FIGURE B-10a. Cut excess carrier tape using razor.

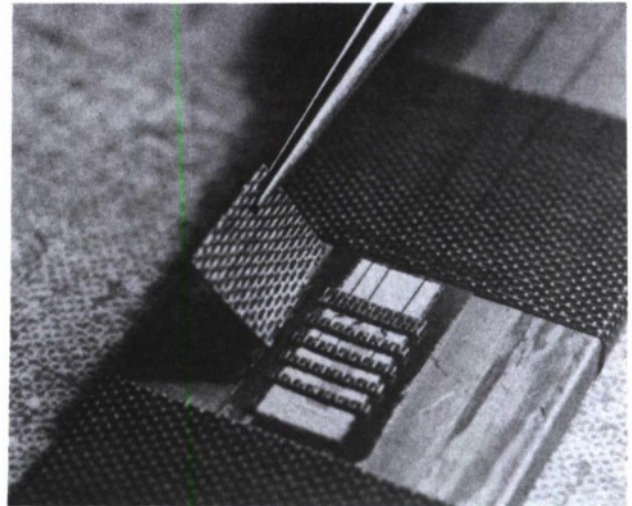


FIGURE B-10b. Remove excess carrier tape.

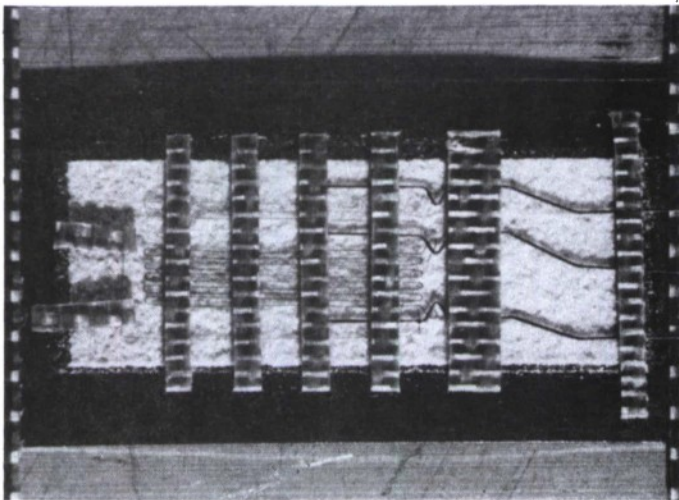


FIGURE B-10c. Apply .030" wide tape bar to cross-over strand on dual element tape.

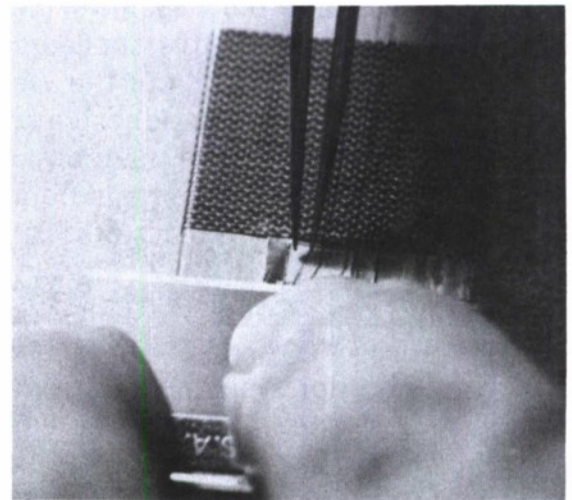


FIGURE B-10d. Place razor flat on tape carrier, press down with finger to prevent movement while bending lead to "S" shape using tweezers.

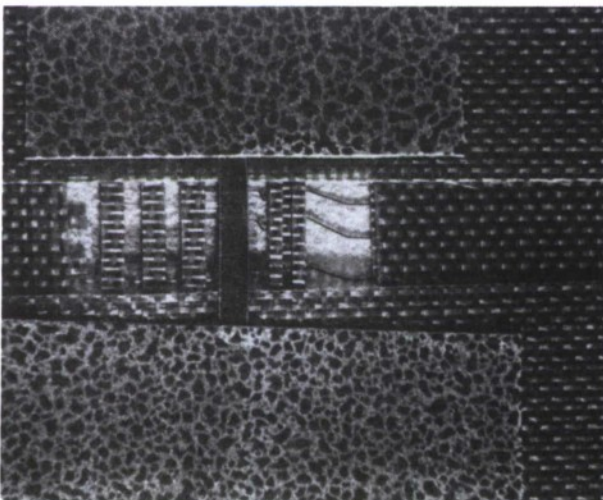


FIGURE B-10e. Place .030" wide x .016" thick spring over loose tape bar and hold in place with aluminum teflon tape.

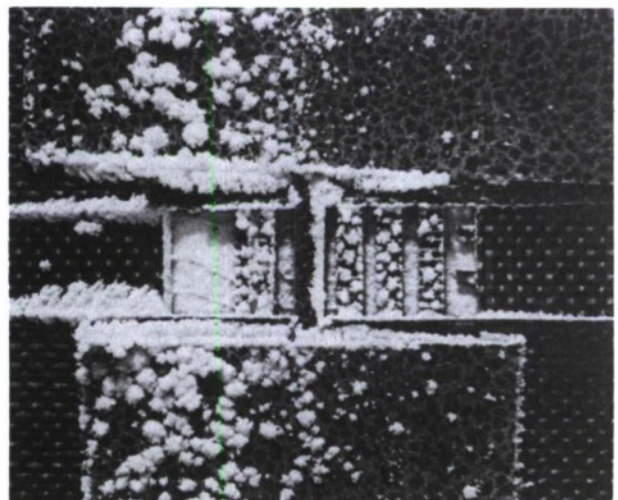


FIGURE B-10f. Flame spray tack coat just enough to hold grid in place.

## FIGURE 10

### **Flame Spray Tack Coat**

Place specimen in vise and adjust position for easy flame spray access. Adjust vortex air cooler to apply a flow of cool air across grid and along tape bars. The nozzle should be 10-20 degrees from horizontal. Use 30 psi pressure.

At a spray distance of 8-10 inches, hold Rokide gun perpendicular to precoat surface and make a rapid pass of about one second with dense cone pattern concentrating spray momentarily on the gage grid area. Move spray pattern in direction of leads as spray is removed from gage. Dwell off the target to allow part to cool. Part should never get warm. Take additional passes and observe coverage. Apply just enough material to hold grid and leads in place. Shut off gun and examine under microscope. Remember, Rokide is very strong. If coverage is sufficient, remove border tape. Remove second layer of tape bars, then remove gage grid tape bars using rolling motion pulling tape at 45 degrees to grid. Do not pull up on tape as this applies very high stresses to the grid wire Rokide bond and might stretch or pull out the wires or leads. After tape removal, inspect under microscope for tape adhesive residue. Brushing with MEK will swell silicone, making it visible. Remove any adhesive using a sharp tweezers or pin or by brushing it out of the gage bond area. Brush off any ceramic particles or blow them away with a blast of clean air (wear goggles).

### **Spray Cover Coat**

Apply border tapes as before except 0.030 inch to 0.060 inch outward of the previous tape location. See Figure B-11. Mount in vise. Apply cooling air, apply final coat slowly, as with the tack coat until the gage is covered. Final installation thickness should be 0.008 inch to 0.010 inch. The coating should never exceed 0.016 inch.

### **Dressing Installation**

Sharp discontinuities are stress risers and are the source of brittle fracture. Dress all installations so the coating layers blend and form a smooth transition between precoat and cover coat. Dress Rokide using an oil-free aluminum oxide stone. A 1/4 inch x 1/4 inch x 4 inch white stone with No. 60 grit is ideal. The edge where the leads come out of the coating may be dressed using a carbide tipped scribe. Press down and at a 30 to 45 degree angle to break off sharp edges.

**Waterproofing** (Same as for ceramic cement)

**Quality Checks** (Same as for ceramic cement)



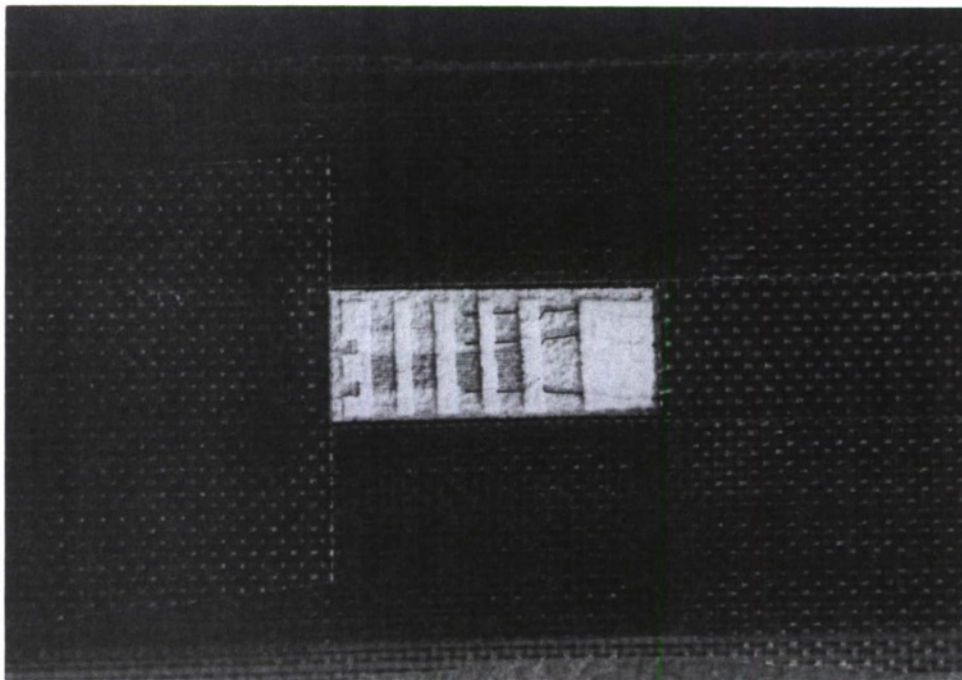


FIGURE B-11. Gage Prepared for Final Coat. Note perimeter tape is about 0.030 inches outward of tack coat position.



## SPECIAL APPLICATIONS

### I. Flame Spray Installation of Flattened Wire Gages

Flattened wire gages are installed using similar procedures to free filament wire gages with special consideration being given to the taping procedures as discussed below.

A round wire end loop is considerably stiffer when it comes to resisting vibration caused by air turbulence from flame spraying. End loops on wire gages can be longer and still be less affected by air turbulence. With flattened gages the tape bars must be placed much closer to the end loops in order to minimize vibration. See Figure 12. Also, the tape bars must be 0.025 inch to 0.030 inch wide and spaced 0.030 inch to 0.040 inch apart. The taping is much more critical than with free filament wire gages.

If the flattened gage end loops stick out too far, flame sprayed material will build up underneath the end forcing the loops to curl up. Build-up under the grid will increase installation thickness. Air vibrations can also cause the gage pigtail to vibrate vertically about mode points A and B as shown in Figure B-13. This can cause early grid wire failure at points A and B, and cause build-up of flame sprayed material under the pigtail, making installations thicker than desired. It may be necessary to add a thin sliver of tape across the end loops and at the pigtail bend to prevent vibration. When the grid is correctly taped, end loop and pigtail vibrations are eliminated, then a very thin installation is possible. Since these gages usually employ inwardly turned pigtails, with lead attachment accomplished out of the grid area, the flame sprayed material need not cover the leads. Spraying is stopped when just enough material is applied to hold the grid in place. Tape bars need not be double taped over the grid because the very sparse tack coat will not entrap adhesive or form crevices at the tape border line. The leads, however, should be double taped.

### Tape Bar Removal

Tape bar removal is accomplished as with free filament gages, by grasping the end of the tape bar with tweezers and rolling the tape off of the grid without pulling upward. If the coating is so sparse as to possibly cause the bonded area to fracture, the adhesive bond may be released by debonding with MEK. This procedure must be done very carefully to avoid silicone contamination. The procedure is as follows: dip a 10/0 artist's brush into MEK and damp dry brush on a gauze pad leaving a small amount of MEK on the brush. While first grasping the tape bar at one end with the tweezers, apply brush to sprayed ceramic near tape. Be careful not to touch the tape. The adhesive will get wet from beneath and release instantly. If the procedure is done carefully, the wetting from beneath will

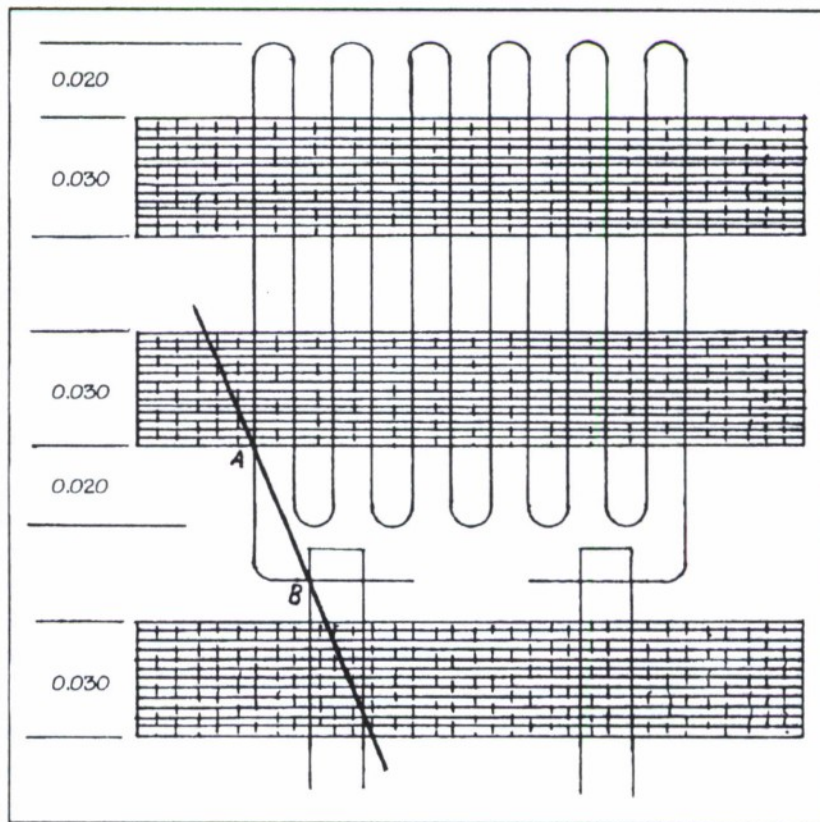


FIGURE B-12. Tape Bar Location for Flattened Wire Strain Gage

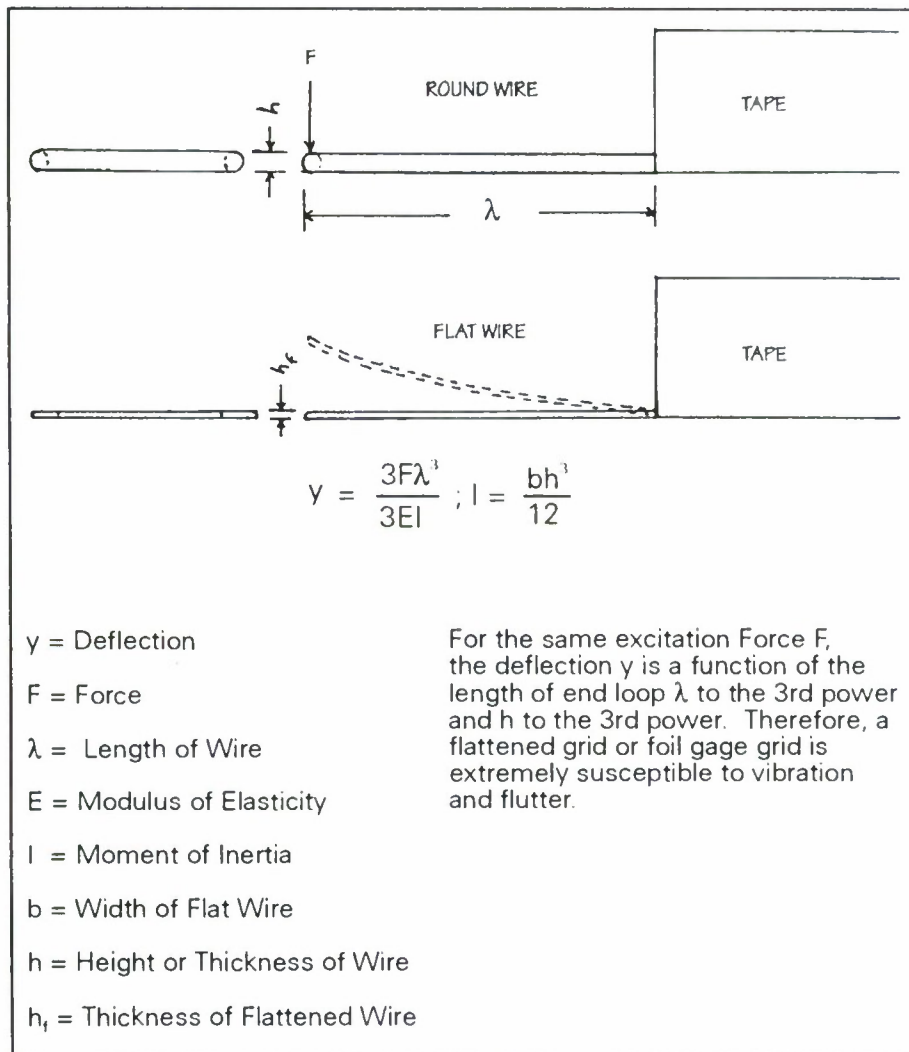


FIGURE B-13. Physical Parameters Affecting End Loop Flutter During Flame Spray



cause instant adhesive release with no contamination. If the adhesive is allowed to float in the MEK, contamination will result.

After tape removal, brush the installation with ample amounts of MEK and look for any silicone residue. Brush residue in one direction out of the grid area. Apply cover coat of flame sprayed material over the grid. Treat the leads as with a free filament gage installation.

## **II. Flame Spray Installation of Foil Strain Gages**

### **Gage Selection**

Most free handling foil gages can not be installed by flame spraying. Only special foil gages having a foil thickness at least 0.0004 inch thick or greater can be applied. The heat of the ceramic particle will melt thinner foil and destroy the gage. The instructions below are for foil gages of 0.0004 inch or thicker.

### **Gage Preparation**

Remove the gage from the packaging and prepare gage by cutting down the end loops on both ends of the gage and trimming the tabs. End loops need not exceed 0.010 inch long and may be cut with sharp scissors while viewing operation under microscope. It is easier to first cut off the leads supplied with the gage, then trim the tabs to about 0.010 inch to 0.015 inch width using sharp scissors under the microscope. Placing the gage on a small platform with the part to be cut off extending beyond the platform makes the job easy.

Excessively long end tabs, besides being difficult to cover, will adversely affect gage factor as well as cause reduced fatigue life due to the stress concentration in the strand/end-tab junction (see theory section in text of report on strain transfer into strain gage). The large solder tab design is also unsuitable for flame spray application, and must be substantially cut down in order to make flame spray coverage possible. Once the tab is trimmed, 0.005 inch chromel wire leads, flattened to 0.0015 inch, can be readily spot welded to the remaining end tab. spot welding should be done using a pencil probe and a flat copper ground plate with the gage placed on the ground plate and the lead placed on top of the pretrimmed tab. Practice welds should be made using the pieces of tab cut off of the gage. After welding, place a tape bar over gage leads to keep leads straight and parallel. Lead length should not exceed 25 mm (1 inch) because longer leads make the gage harder to handle.

### Gage Application

Place the gage on the precoat and align grid with alignment marks. If the grid is slightly distorted, it can be nudged into a straight and parallel grid pattern position using tweezers. With the grid strands straight and parallel, tape the grid to the precoat using 0.8 mm (0.030 inch) wide tape strips. The spacing between tape strips should also be about 0.8 mm (0.030 inch). Place a strip of tape over the end of the end loops about 0.1 mm (0.004 inch) inward to prevent the end loop from vibrating in the turbulent air. The leads should be bent into convoluted "S" pattern using tweezers, (as with free filament gage installations), and taped in place with 1.2 mm (0.050 inch) wide tape strips. The leads should be double taped using 1.5 mm (0.060 inch) wide tape strips. Apply a single layer of tape around perimeter of installation and inspect under microscope to check for bond.

Mount the specimen in the flame spray booth vise and apply vortex cooling air. Flame spray as with a free filament gage with just enough material applied to hold the grid in place. Do not fully cover grid. Inspect again under microscope and, if necessary, flame spray again until just enough material is applied to hold the grid in place. See Figure B-14. Inspect under microscope; if coverage is adequate, remove perimeter tape, then remove tape strips over grid using a rolling motion. Do not lift up on tape. If necessary, apply MEK using the same procedure as with flattened wire gages to debond tape strips. Inspect for silicone tape residue. Remove residue using MEK and artist brush. After thorough drying, reapply tape around perimeter about 0.8 mm to 1.5 mm outside of the previous perimeter tape location. Apply flame sprayed cover coat to gage grid. Then place a piece of teflon fiberglass tape over gage grid and complete lead installation as with free filament gage.

### III. Composite Installations

Composite installations employ a combination of flame spray and ceramic cement techniques. This usually consists of a flame sprayed precoat and a ceramic cement tack coat, followed by a flame sprayed cover coat. The technique is especially useful for gage attachment to curved surfaces where flame sprayed particles can get under the shading tape layer and bond to the adhesive. See Figure B-15. In this type of application, the gage is positioned on the flame sprayed precoat and tack bonded with ceramic cement. The installation is air dried, cured 1/2 to 1 hour at 200 - 225°F, and 5 to 10 minutes at 350°F. Cool to room temperature, remove the tape carrier, remove any tape residue using MEK, then cure cement for 1/2 to 1 hour at 600 - 650°F. Upon cooling to room temperature, mask perimeter of gage bond area and apply cover coat of flame sprayed material.

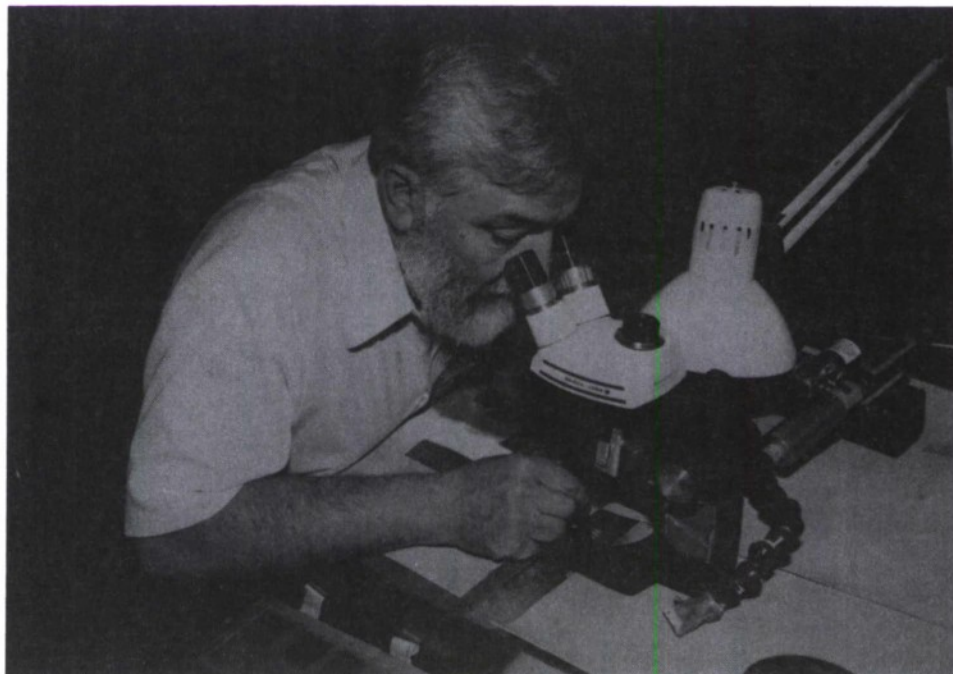


FIGURE B-14. All gage installation work should be performed under microscope.



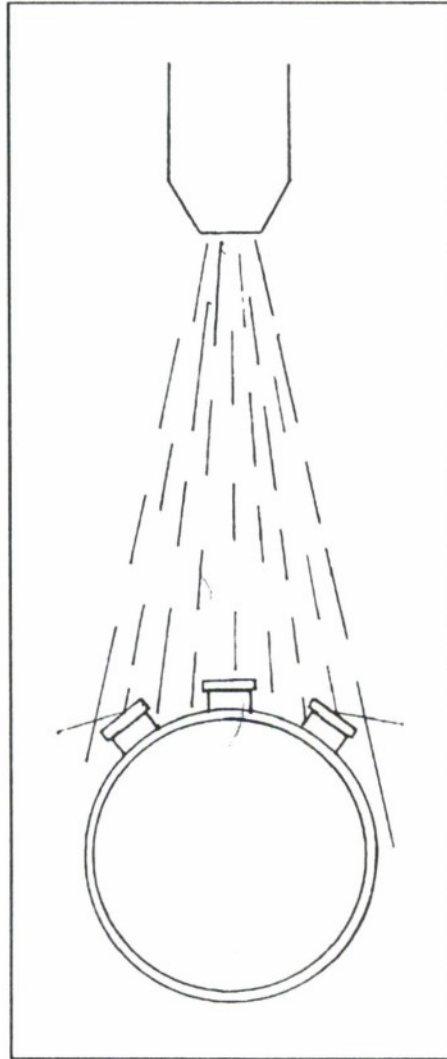


FIGURE B-15. Flame spraying curved surfaces is difficult.

#### IV. Powder Flame Sprayed Installations

Powder flame sprayed installations were made during this program using the Hitec CR-1000 Mini-gun flame spray system. The system is identical to that described for nickel aluminide applications but the operational parameters are different. The settings for spraying aluminum oxide are:

Oxygen pressure:	20 psi
Acetylene pressure:	6 - 10 psi (start at 7.5 psi)
Oxygen flow:	6.5 points
Acetylene flow:	6.0 points
Powder flow:	5.0 points
Spray distance (nozzle to specimen):	6 inches
Vortex cooling air pressure:	30 psi

The procedure for powder flame spray installations are similar to those used for Rokide with special considerations described below. The process is much hotter than Rokide and the coating passes must be made extremely fast. The quickness of the coating pass is difficult to judge and each technician should develop his technique while measuring grid temperatures during flame spraying. The HFP gage type is extremely sensitive to temperature, producing about 70 microstrain per degree Celsius and is useful for developing technique. Solder lead wires to the gage and connect it to a strain indicator or X-Y plotter, and measure gage output during flame spraying. The best way to do this is to set the X-Y plotter with the X-axis on a time scale, about 50 seconds per centimeter, with the gage output on the Y axis. The output of the gage should not exceed the equivalent of 100°C temperature rise. Since only 50% of the gage grid is exposed, this is equivalent to about 200°C wire temperature. If this temperature is exceeded, adjust spraying using quicker passes and continue measuring temperatures until the grid temperature is held to an acceptable level.

The gage grid is a very small mass and responds to changes in temperature extremely fast. As the grid heats up it also expands. This expansion produces a columnar force which, since the ends of the wire are held down, will cause the grid to pop up and vibrate in the air turbulence. The end result is a build-up of flame sprayed material under the grid wire. The thickness of material built up under the grid is mainly a function of the thermal expansion coefficient and the temperature of the grid wire. Calculations indicate that a 0.1 mm to 0.2 mm of build-up is easily achievable.

Other problems caused by excessive heat-up of the wire are:

1. Oxidation of the grid wire
2. Annealing the grid wire or altering the heat treatment

### 3. Melting the wire

Once the material builds up under the grid wire, it cannot return to its original straight position upon cool down. Instead it cools in a state of residual tension. This reduces the strain limit of the gage installation as well as the fatigue life. With a grid located well above the precoat, the gage factor is also affected. The resulting thicker installations have a reduced strain limit and less thermal shock capability.

With the double tape system and vortex cooling, extremely quick spray passes will result in thin gage installations. See Figure B-16. The part should never get warm. Proceed with installations using the same methods as for Rokide.

### **V. Flattened Wire Gages, Ceramic Cement Bonded**

Flattened wire gages are more suitable for ceramic cement application because the problem of air turbulence from flame spraying is eliminated. These gages can be bonded over ceramic cement or Rokide precoat. Rokide precoat is often used because it is much easier to apply a uniform precoat with Rokide, especially on curved surfaces.

Position gage on precoat and lay in place. Because there is no air turbulence to worry about, the grid can be held in place using a wire rod or ribbon, to avoid tape adhesive contamination problems. A 0.020 inch diameter wire, prebent to conform to surface curvature is placed over grid and held at the ends with tape. Press on wire to be sure grid is in intimate contact with precoat. Leads may be held with wire or with tape as shown in Figure B-17.

Apply cement to gage end loops and on leads. Some cement thinning may be required because the Rokide precoat quickly takes the liquid out of the cement. Be sure not to add excessive liquid. It is undesirable to allow liquid to migrate under tape (if tape is used). Care should be taken when applying Rokide precoat to make it as thin as possible. The precoat should be gray, not white. Excessive precoat thickness absorbs liquid from cement, causing it to ball up rather than spread.

Apply just enough cement to cover grid end loops and leads. Air dry as required (1/2 - 6 hrs at room temperature). Cure for 1/2 to 1 hour at 200-225°F, followed by 5 to 10 minutes at 350°F. Cool. Remove tape and hold down wire. Cure for 1/2 to 1 hour at 600 to 650°F, and 2 hours at 800 to 850°F (if tape is used). Apply cover coat of cement to exposed grid and lead wires up to level of grid wires. Allow to dry. Apply final cover coat of cement over entire gage and lead area blending cement to a uniform cover coat. Air dry as required. Cure for 1/2 to 1 hour at 200°F to 225°F, 1/2 to 1 hour at 400°F and 1/2 to 1 hour at 600 to 650°F.



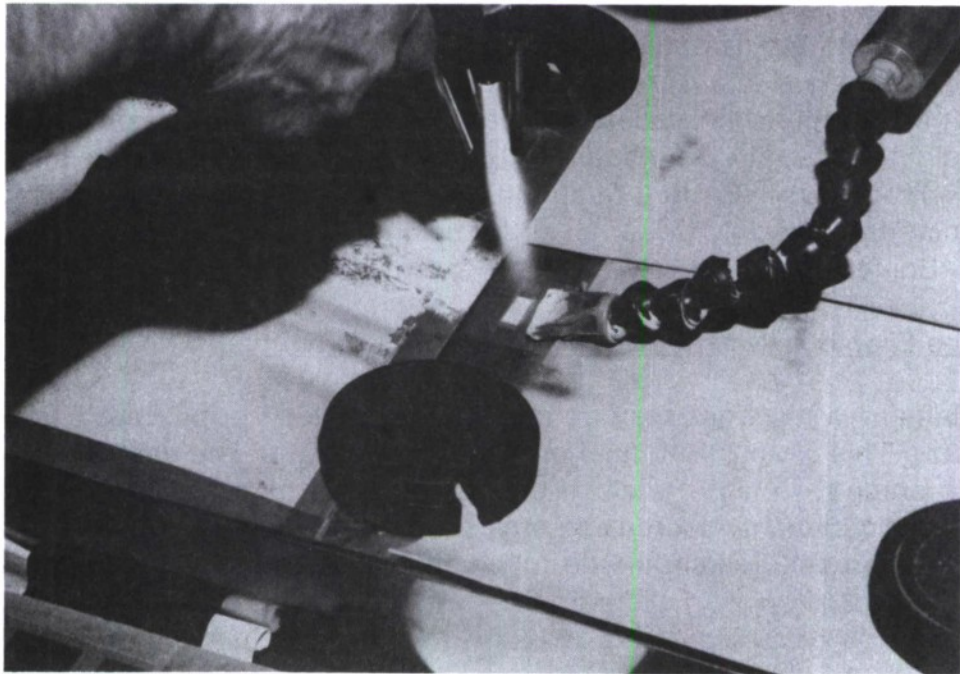


FIGURE B-16. Vortex air is applied to gage during flame spraying.

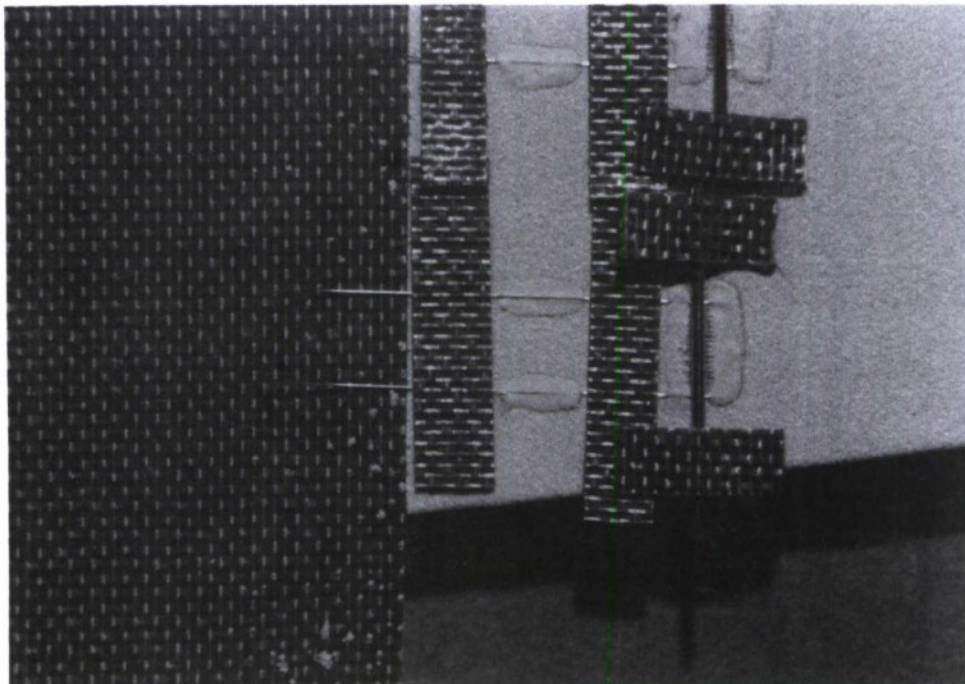


FIGURE B-17. Flattened Wire Gage Installed Over Rokide Precoat. Note wire holding grid in contact with precoat. Cement is applied to gage end loops and leads.

Note that once tape is applied to the precoat, the installation should be carried out to the point where tape is removed. Do not permit the tape to stay in contact with precoat any longer than necessary, or the silicone adhesive from the tape will contaminate precoat. See Figure B-18. Where the tape was left on over a weekend, and even at room temperature obvious contamination resulted. This contamination can only be removed by baking in an oven at 800 to 850°F for 2 hours. Solvents will not remove such extensive contamination.

#### **VI. Free Filament Wire Gages, Ceramic Cement Bonded**

Free filament wire gages may be bonded over Rokide or powder flame sprayed precoat. This is an often used procedure because of the ease in applying a uniform precoat. Also, the electrical properties of high purity Rokide are better than ceramic cement, decreasing errors in static strain measurement. The same installation procedures should be followed as with ceramic cement precoat.

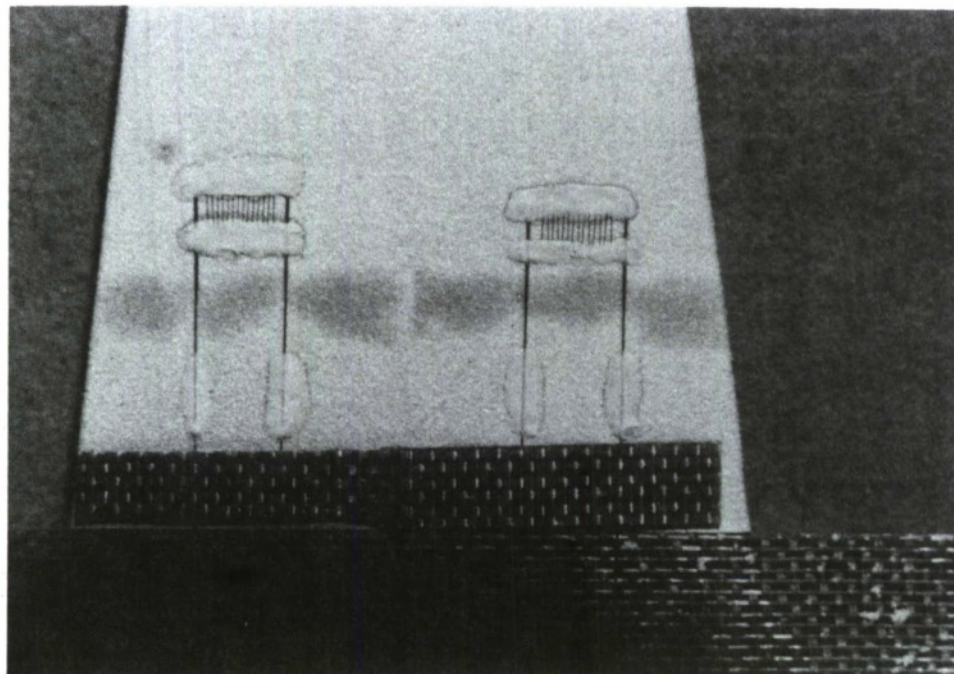


FIGURE B-18. Hold-down wire and tape are removed from gage. Note clean area where grid was held down with wire and contaminated area caused by tape. Contamination occurred after a few days at room temperature.